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TAMPERE UNIVERSITY OF TECHNOLOGY

IKECHUKWU OKONKWO

MOMORDICA ANGUSTISEPALA AS REINFORCEMENT IN POLYMER COMPOSITES

Master of Science thesis

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ABSTRACT

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The use of natural fibers as reinforcements for polymers has been receiving great attention in recent times. This is because natural fibers are cheap, abundant and environmentally friendly. Momordica Angustisepala is one of such fibers, and this research explored its potentials for use as reinforcement in polypropylene. The effects of pretreatment were also examined to determine its efficacy in improving fiber-polymer adhesion which has been noted as a persistent problem in similar composites. Fiber treatments were performed using sodium hydroxide, potassium hydroxide, silane, dicumyl peroxide, maleic anhydride, and sodium hypochlorite. The raw and treated fibers were studied to determine the tensile properties as well as the density and water absorption. Selection of the best fiber treatment was made using Minitab's Central Composite Design, (CCD). Composites were produced with the fibers and polypropylene, using compression moulding. Tests were performed to determine the mechanical properties of the composites, the density and the water absorption. The morphology of the fibers and composites were also studied using scanning electron microscopy (SEM).

The moisture content of the fiber at room temperature was determined to be 7.2% and its water absorption was 95%. Various chemical treatments decreased the water absorption of the fiber bundles to 80% - 40% range and decreased bulk density in most cases from 1.11g/cm^3 to 1.1g/cm^3 – 0.6g/cm^3 range. Fiber treatment with 5% NaOH for 1 hour was selected for composite production since it offered fibers with best tensile strength and modulus as well as moderate water absorption and density. Results of composite tests showed a decrease in most mechanical properties, except Young's modulus which was increased by 2.0%. Tensile strength decreased by 24%, but the tensile strength and Young's modulus of the treated-fiber composite were higher for than that of the raw-fiber composite. Flexural strength and modulus decreased by 7.0% and 18.7% respectively. Impact strength was reduced by 65.9%. The composites have higher density and water absorption than the original polymer. However, the density of the composite was decreased by 4.7% when treated fibers were used instead of raw fibers, and water absorption was similarly decreased by 2.3%. From SEM images, better adhesion was observed in the fiber-matrix interphase of the treated-fiber composite.

Therefore, pretreatment was effective in improving the tensile properties of treated-fiber composites when compared to raw-fiber composites. Similarly, chemical treatments also helped to cushion the effects of fiber-addition on the density and moisture resistance of the composites. However, reinforcement was only achieved in the stiffness of the composite since other mechanical properties of the polymer were higher than that of the composite. Future researches may determine the effects of using higher weight fractions of fiber, or using other processing methods, for producing the composite.

PREFACE

This thesis is a research work was performed in the laboratories of Materials Science department at Tampere University of Technology. It is the culmination of my Masters' program at the university. This work took me though many laboratories in the fields of polymer and fiber engineering, and offered me a lot of experience.

I want to acknowledgement my supervisors Dr. Marja Rissanen and Prof. Jyrki Vuorinen for kindly supporting this work.

I also want to acknowledge my parents, Patrick and Caroline, and my sisters for supporting my graduate studies in Finland, and for their prayers.

Tampere, 23.3.2015

Ikechukwu Okonkwo

CONTENTS

LIST OF FIGURES

LIST OF TABLES

.

LIST OF ABBREVIATIONS

PP	*polypropylene*
LDPE	*low density polyethylene*
HDPE	*high density polyethylene*
SEM	*Scanning electron microscopy*
CCD	*Central composite design*
RSM	*Response surface methodology*
M.A.	*maleic anhydride*
NaOH	*Sodium hydroxide*
KOH	*Potassium hydroxide*
silane	*Methacryloxypropyl-trimethoxysilane*
DCP	*dicumyl peroxide*
NaOCl	*sodium hypochlorite*
OH group	*Hydroxyl group*
RH	*Relative humidity*
mm	*millimeter*
µm	*micrometer*
KV	*Kilovolts*
MPa	*Megapascals*
GPa	*Gigapascals*
g	*gramme*
mm/mm	*millimeter per millimeter*
J/mm^2	*Joules per squared millimeter*
g/mm^3	*grammes per cubed millimeter*
g/cm^3	*grammes per cubed centimeter*
v-%	*volume percent*
w-%	*weight percent*
%	*percentage*
hrs	*hours*

1. INTRODUCTION

Polymers are exceptionally useful materials that have found application in numerous applications. They are mainly sourced from by-products of crude oil, and also from other materials such as coal and wood. Polymers possess unique properties that have enabled them to serve as replacements for traditional materials like metals and ceramics in diverse applications. Some of these properties are inherent e.g. light weight and high specific strength, while others result from the incorporation of additives in polymers –oxidation resistance, fire resistance, etc.

In spite of many laudable features of polymeric materials, some of their properties, especially mechanical properties, remain relatively low when compared to metals and ceramics. For instance, the ultimate tensile strength and tensile modulus of polymers are considerably lower than those of metals. This discrepancy had given rise to the use of reinforcements in polymers in order to boost these properties. Initially, reinforcement materials for polymers are usually glass fibers, carbon fibers and synthetic fibers like aramid. Recently however, natural fibers have generated much interest as good reinforcement for polymers. Flax, hemp and many other types of natural fibers have been used. Momordica Angustisepala, a bast fiber, is also a potential reinforcement that could be used in polymers, in the production of composites.

Composites are materials comprising two or more materials whose properties complement each other. There are metal matrix composites, ceramic matrix composites and polymer matrix composites. Polymer composites are the combination of polymers and other materials to obtain materials with improved properties. Polymers can be thermosets or thermoplastics. Thermosets, unlike thermoplastics, cannot be melted and remoulded after the initial production; thermoplastics can easily be recycled or remoulded. Polyester, epoxy and vinylester are commonly used thermosets for composite production, while polyethylene, polypropylene (PP) and polyvinylchloride (PVC) are commonly used thermoplastics.

A composite consists of the matrix and the reinforcement. In polymer composites, the polymer is the matrix. The matrix material binds the reinforcements and gives form to the product. It transfers the load or stress to the stronger reinforcing material. Reinforcement materials in polymers are fillers and fibers. Fibers can be short, long or continuous. Continuous fiber reinforcements are usually obtained from carbon or aramid fibers, and are primarily employed in the manufacture of composite parts for high end products such as

aircrafts. Short fibers are primarily obtained from chopped fibers from glass or milled carbon fibers. Long fibers offer greater strength and stiffness than short fibers.

Manufacturing methods for polymer composites include hand layup, spray layup, pultrusion, resin transfer moulding, compression moulding, reaction injection moulding and many others. The manufacturing method to be adopted depends on the type of fiber as well as the type of polymer. Fiber type and fiber distribution greatly influences the mechanical properties that can be achieved in the produced composite. Other important factors that influence the properties of fiber-reinforced polymer composites include the fiber geometry, fiber volume (or weight) fraction and fiber-matrix interfacial shear strength [1].

There is increasing interest in the use of natural fibers as reinforcements in polymer composites. This rising interest is not unrelated to the fact that their alternatives –synthetic fibers, are expensive and are neither biodegradable nor environment-friendly. While synthetic fibers have impressively high tensile strength and modulus, they can only be used in such industries as automobile or aerospace, due to their high costs. An exception is glass fiber which is cheaper than carbon and aramid fibers, and is used in more general application.

On the other hand, natural fibers or fibers derived from lignocellulosic plant products, are quite cheap, biodegradable and environment-friendly. They are abundant and easily accessible in various climates, and can be easily and safely disposed of at the end of their lifecycle. They are naturally produced unlike synthetic fibers, and therefore do not require energy consumption at the fiber-formation stage. Other advantages of natural fiber as reinforcements are their light weight and high specific strength [2] or strength per unit weight, due to their relatively low density.

Natural fibers possess properties and geometry that vary according to the source plant, growing climate, plant age, harvesting, storage, and processing conditions. Some common natural fibers include hemp, kenaf, flax, henequen, pineapple leaf, jute, and bamboo fibers. The properties of natural fibers used as reinforcements influence the properties of the resulting polymer composites. Hence, polymer composites containing natural fibers have light weight, and possess good specific strength. They also positively influence most other properties of the original polymer. The properties of such polymer composites are however, also dependent on the extent of fiber-matrix adhesion or the ability of the fibers to effectively bond with the polymer matrix. Fiber-matrix adhesion is a common problem in natural-fiber-based polymer composites, arising from the incompatibility of the fiber surface with that of the polymer matrix.

Natural fiber surface is not very compatible with the surface of polymers. This problem reduces the mechanical properties of the composite; premature failure usually occurs at the weak interface between the fibers and the polymers. This tendency is remedied by surface

modification of natural fibers prior to use in polymer composites. Surface modification entails treatment of the fibers with various chemicals. These chemicals typically react with the OH group of the cellulose compound, creating reactive sites that ultimately bonds with the polymer. Such chemical reactions is also known to address another major issue of using natural fibers in composites –their hydrophilic nature which makes them absorb enormous amount of water [3]. Surface treatment increases moisture or water

This study aims to explore the potentials of a natural fiber –Momordica Angustisepala –for use as reinforcement in polymer composites. This fiber is quite commonly found in the forests of West Africa. The source plants grow naturally, and do not require any tending or caring. This makes them quite economical and cheap. A cursory observation and hand testing also revealed that this fiber is relatively stronger than most fibers of the same class. This makes them potentially useful for engineering application. The study will determine how the fiber affects some properties of polypropylene. Fiber and composite properties will be studied; the fibers will be treated with several chemicals, and the effects on fiber and composite properties due to the surface treatments will be examined.

The research will characterize the fibers and obtain values for density, water absorption, tensile strength and tensile modulus. Tensile, bending and impact properties will be obtained for the polypropylene composite. The research will also capture the morphological images of the fibers and composite.

The previous literature in the area of polymer reinforcement with natural fibers will be reviewed in the chapter two of this work. Chapter three will describe the materials and methodology employed in the research. In the fourth chapter, the results obtained from the tests will be presented and analyzed. Finally, a conclusion of the findings and efficacy of the whole research will be given in the fifth and final chapter.

2. NATURAL FIBER BASED COMPOSITES

Rising interest in natural fibers as effective reinforcements for polymers has continued to generate many researches and findings. Many studies has been conducted on these fibers as well as on the composites made from them. Possible uses have been suggested and some have already been implemented. This chapter will endeavor to review some of these researches and findings, especially those relating to the physical and mechanical properties of natural fibers and their polymer composites.

2.1. Natural fiber structure and properties

Natural fibers can be obtained from plants, animal or mineral sources.

Plant fibers can be categorized into bast fibers, leaf fibers, fruit fibers, grass fibers, straw fibers, wood fibers, root fibers and others. A schematic representation of this classification is shown in figure 2.3.

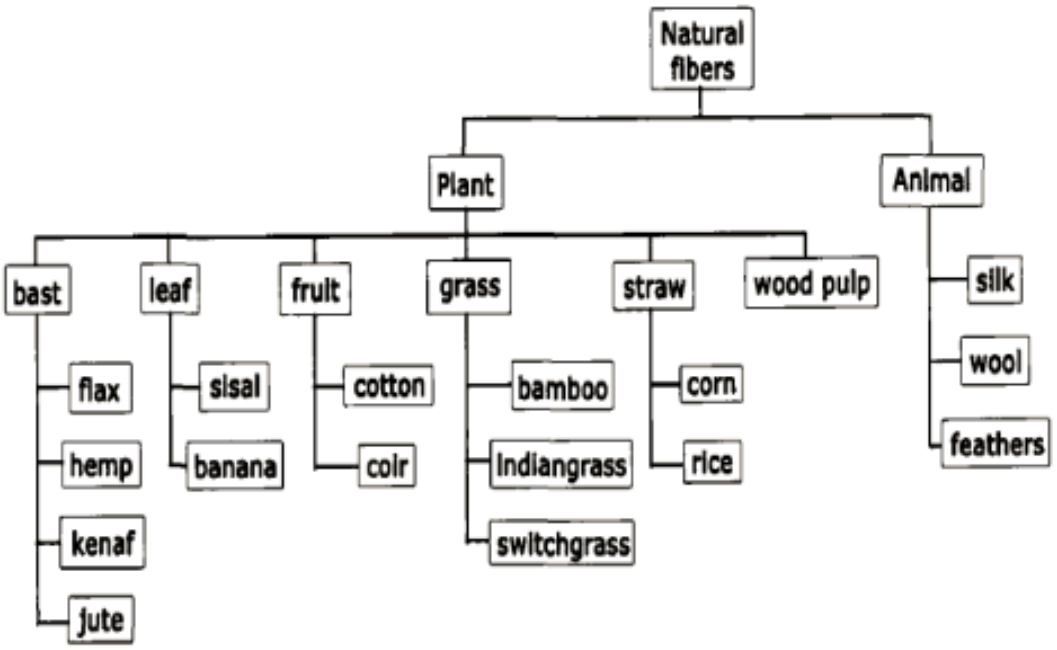

Fig 2.1. Classification of plant fibers [8]

Most reinforcements are however sourced from bast fibers due to their relatively higher stiffness. Plant fibers are composed of these substances: cellulose, hemi-cellulose, lignin,

pectin, wax, water soluble and water [4]. These fibers are essentially a composite of mostly cellulose fibrils bonded by lignin acting as the matrix. A fiber strand consists of several layers of walls enclosing a middle section. This middle section contains the microfibrils that comprises of long chains of cellulose molecules. These microfibrils have helical structure whose angles (θ) with the fiber axis vary in different fibers [5]. These features are depicted in figures 2.1 and 2.2 below.

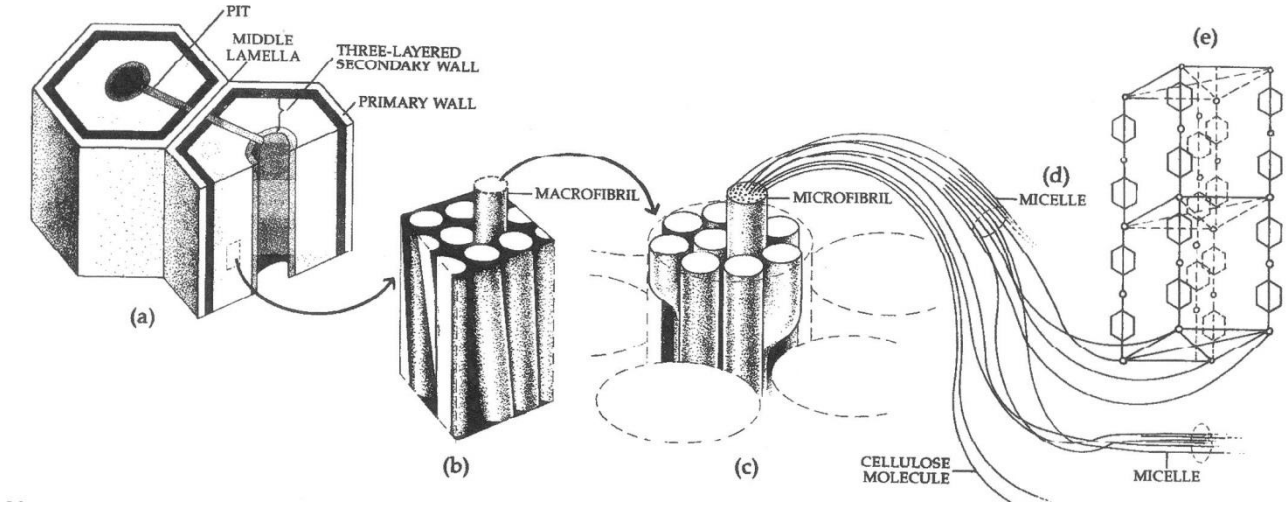

Fig. 2.2. Structural representation of a plant fiber [6]

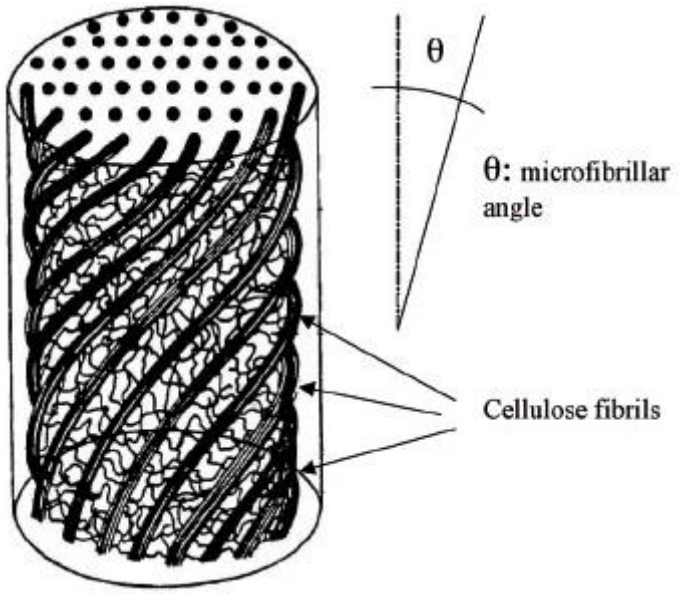

Fig. 2.3. Helical structure of microfibrils [7]

Plant fibers, especially bast fibers, possess physical and mechanical properties that make them suitable as replacements for traditional reinforcement fibers in composite production. This is especially the case for specific mechanical properties because of the low weight of

natural fibers. There are however some variations in the reported properties of particular fibers due to a number of factors. Some of these are: differences in the fibers chemical composition and structure (microfibrillar angle, crystallinity, defects) due to growth-stage environmental conditions; use of different testing methods and environmental conditions (relative humidity, temperature, speed loading, number of sample tested); different scales (single fibers or fiber bundles) of measurements -testing performed at the bundle scale encounter slippage effects of the fibers relative to each other in the middle lamella [9]. Table 2.1 below shows a range of values for properties of some fibers.

Table 2.1. Properties of different fibers [9].

Fiber	**Density**	**Young's modulus (GPa)**	**Tensile strength (MPa)**	**Elongation at break (%)**
Flax	1.54	27.5-85	345-2000	1-4
Ramie	1.5-1.56	27-128	400-1000	1.2-3.8
Hemp	1.47	17-70	368-800	1.6
Jute	1.44	10-30	393-773	1.5-1.8
Sisal	1.45-1.5	9-22	350-700	2-7
Coconut	1.15	4-6	131-175	15-40
Cotton	1.5-1.6	5.5-12.6	287-597	7-8
Nettle	1.51	24.5-87	560-1600	2.1-2.5
Kenaf	1.2	14-53	240-930	1.6
Bamboo	0.6-1.1	11-17	140-230	-
E-glass	2.5	70	2000-2035	2.5
Carbon	1.4	230-240	4000	1.4-1.8

Natural fibers contain a high percentage of cellulose -a semicrystalline polysaccharide made up of D-glucopyranose units linked together by b-(1-4)-glucosidic bonds [10]. These contain a high number of OH-groups that makes the fibers hydrophilic. This property of fibers reduces their compatibility with polymers which are hydrophobic. The poor compatibility is due to inadequate interfacial adhesion and low moisture resistance of the fibers [11]. The hemicellulose components of natural fibers are the lead cause of their hygroscop-

ic nature since hemicellulose has partly soluble open structure containing many hydroxyl and acetyl groups [12]. Surface treatment of fibers helps to improve their quality and potential for use as reinforcing materials. Surface treatment with chemicals modifies the fiber chemical structure making it more reactive and also less hydrophilic [13–17].

2.2. Effects of fiber surface treatments

Fiber surface treatment serves several purposes including fiber-matrix coupling in composites. Chemicals used as coupling agents generally perform two roles; they react with the hydroxyl groups of the cellulose and also react with the functional group of the matrix during the formation of the composite. There are several treatment types including alkaline, silane, acetyl, benzoyl and maleic anhydride and permanganate treatments.

The most common chemical treatment is performed using alkalis in a process known as mercerization. The process involves the breaking of hydrogen bonding and consequently causes the roughening of fiber surface. Alkali treatment results in the removal of the superficial lignin, wax and oils on the cell wall of bast fibers, and also breaks the cellulose chains and frees short length crystallites [18]. The hydroxyl groups of the natural fiber cellulose are ionized to alkoxides on the treatment with sodium hydroxide (NaOH) solution [19]. Treatment is usually performed by timed immersion of fibers in solutions of specific concentrations. For instance, jute and sisal fibers have been treated with 5% aqueous NaOH solution at various times ranging from 2 to 72 hours, and at room temperature [14, 15]. For flax fibers, modification was achieved by immersion in 2% alkali solution for 90 seconds at temperature and pressure of 200^{o}C and 1.5 MPa, respectively [20]. Studies have shown that alkali treatment increases fiber surface roughness which causes improved mechanical interlocking, and also increases number of possible reaction sites by exposing more cellulose on the fiber surface [21]. The treatment has been shown to improve fiber strength and stiffness of flax fibers [22], and offered 30% increase in the strength and modulus of flax fiber–epoxy composites [17]. It also greatly improved the mechanical, impact fatigue and dynamic mechanical behaviors of several fiber-reinforced composites [16, 23, 24]. 4% NaOH [24] and 5% NaOH [25] has been reported as the best concentrations for sisal fiber treatment to obtain optimal mechanical properties in the reinforced composites. Higher concentrations could lead to fiber degradation from excess delignification, and consequently, diminished mechanical properties of the composites [25].

Another commonly used chemical for fiber treatment is silane. Silanes contain alkoxy groups which react with water molecules to form silanols. These silanols react with the hydroxyl group of the fiber to form stable covalent bonds with the cell wall, the product of which are adsorbed to the fiber surface [19]. These covalent bonds are extended from the treated fibers to polymer matrix in a composite, in a network-like structure. Initially, silanes were mostly used in glass fiber treatment [26–29] but are now also used for natural

fibers due to observed efficacy. Several processes have been adopted for this treatment method. Flax fiber was treated for 2 hours with 1% concentration of 3-amino propyltrimethoxy silane in in 50/50 acetone and water solution [17]. Sisal fibers was treated by immersing them for 5 minutes in a solution of 2% aminosilane in 95% alcohol, in 4.5–5.5 pH, then air dried for 30 minutes [30]. Henequen fibers were treated with 0.033% silane concentration in water/ethanol solution and it improve the tensile strength of the resulting composite [31]. Oil palm fibers were also treated with 1% silane concentration in water/ethanol solution and the thermal properties of the composite made from the fibers were improved due to the treatment [19].

Researchers have also used acetylation to treat and modify natural fiber surface before being used in polymer composites [30, 32-34]. The acetylation process involves the use of acetic anhydride on the cellulose such that the hydroxyl groups on the cellulose chain are replaced by the acetyl groups. The process reduces the water absorption potential of natural fibers, giving them the hydrophobic property suitable for fiber reinforcements in polymer composites. Sisal fibers have been treated by this method [25]. The fibers were pretreated in 5 and 10% sodium hydroxide solution before acetylation. For acetylation, the fibers were immersed for 1 hour at 30^{o}C in glacial acetic acid and then subsequently immersed for 5 minutes in acetic anhydride containing one drop of concentrated sulphuric acid (H_2SO_4). Sisal fibers have also been treated in 18% NaOH solution and then in glacial acetic acid, and later in acetic anhydride mixed with two drops of concentrated H_2SO_4 for 1 hour [35]. Consequently, the fiber surface became rough and exhibited better mechanical adhesion with polymer matrix in a composite.

Hydrophobicity has also been improved in natural fibers through benzoylation, a treatment type in which benzoyl chloride is used to modify fiber surface. Sisal fibers have been surface-modified by treatment with 10% sodium hydroxide and benzoyl chloride solution for 15 minutes [36] while interfacial adhesion between flax fiber and polyethylene has been improved using the same process [37]. There was first a pretreatment with NaOH in order to activate the hydroxyl groups of the cellulose and lignin in the fiber, before the main treatment. Excess benzoyl chloride on the fiber surface was later removed by immersing the fibers in ethanol and then washed with water and oven-dried for 1 day at 80^{o}C.

Natural fibers have also been modified using acrylic acid or using acrylonitrile in a process known as acrylation. This process has been used to treat oil palm fibers. The fibers were pretreated for about 30 minutes with 10% NaOH, and then treated for 1 hour with different concentrations of acrylic acid at 50^{o}C, then rinsed with alcohol and dried [38, 39]. It was noted that this treatment did not increase the tensile strength of oil palm fiber and polyethylene composite. However, for flax fiber and high density polyethylene (HDPE) composite, the tensile strength and Young modulus were increased while the water absorption was reduced [40].

Other treatment methods that have improved fiber-matrix adhesion and composite mechanical properties include maleated coupling agents or maleic anhydride [41–44], permanganate treatment using potassium permanganate ($KMnO_4$) in acetone solution [10, 16, 23, 48, 49, 54], peroxide treatment using organic peroxides such as benzoyl peroxide (BP) or dicumyl peroxide (DCP) in acetone solution [32, 16, 38, 39]. Others are isocyanate treatment using toluene diisocyanate or its derivatives [32, 12, 16, 34, 45], stearic acid treatment [32, 46] and sodium chlorite treatment [47].

2.3. Natural fibers as reinforcements in polymer composites

Natural fibers generally increase the mechanical properties of polymers to some extent. A number of polymers have been combined with various natural fibers but the most commonly studied are epoxy, polyester, vinyl ester, polypropylene (PP) and high- and low-density polyethylene (HDPE and LDPE). The important factors that influence the properties of the resulting composite include the type of fiber, the type of polymer, the fiber-matrix ratio, the fiber-matrix adhesion and the processing method.

Various researches have reported different levels of changes in properties of polymer on being combined with natural fibers. For mechanical properties such as tensile and flexural properties, there have been increases in such properties of the polymer provided sufficient quantities of fibers have been added to produce the composite. For instance, addition of short banana-stem fibers increased the tensile strength of epoxy by 90%, the flexural strength by 38%, the Young's modulus by 36% and the flexural modulus by 17% [48]. Reinforcement with 15 volume percent (v-%) banana-stem fiber has also been shown to increase the tensile strength of polypropylene by 34%, the flexural strength by 48%, the Young's modulus by 83% and the flexural modulus by 125% [49].

Quantities of fiber used are usually measured in volume or weight percent (w-%) depending on researchers' preference. For both scales, the ratio normally used range from 10% to around 60%. The weight scale is more commonly used in engineering due to its accuracy and ease of measurement. Higher fiber ratios seem to yield more increase in mechanical properties, until an optimum value is reached. For composite studies, the mechanical properties usually measured include tensile strength, Young's modulus, flexural strength and flexural modulus. Studies have shown that reinforcement with 30 w-% coconut fibers improved the tensile strength of epoxy by 307% and the Young's modulus by 55% [50]. The flexural strength was also increased by 39% [50]. Similarly, addition of 30 w-% bagasse fibers was shown to increase the flexural strength of epoxy by 23% [51]. It has also been reported that the tensile strength of polyester was increased by 152% and the flexural strength by 315% when reinforced with 65 w-% bagasse fiber [52].

Researches have also shown that plant fibers are suitable for reinforcement of both thermoplastics and thermosets. Most common polymers of both categories have been combined with such fibers and changes in properties have been recorded in all cases. As have been earlier indicated, commonly used thermoplastics are PP, LDPE and HDPE while commonly used thermosets are polyester, epoxy and vinyl ester. Composite production with 30 w-% sisal fiber increased the tensile strength of LDPE by 60% and the Young's modulus by 458% [53]. It also increased the tensile strength of polypropylene by 27% and the Young's modulus by 140% [53]. Similarly, addition of 60 volume percent jute fibers increased the tensile strength of polyester by 900% and the Young's modulus by 775% [54]. In another study, 60 w-% jute fibers increased the tensile strength of polypropylene by 120% and the flexural strength by 100% [55].

The extent of changes recorded for properties of polymers usually have a wide variation among researchers as there is no definitive range of property values for polymer composites reinforced with natural fibers. The percentage change could range from single digits to hundreds, depending on the earlier mentioned factors: fiber type, polymer type, fiber-matrix ratio, fiber-matrix adhesion and processing method. For instance, reinforcement with 40 w-% pineapple-leaf fibers increased the tensile strength of polyester by 176% and the Young's modulus by 335% [56]. The same study also showed that 30 w-% of this fiber in polyester composite increased the flexural strength by 13% and the flexural modulus by 105%. In a related research, 20 v-% pineapple-leaf fibers was used to increase the tensile strength of polypropylene by 43% and the Young's modulus by 36%, while 10 v-% increased the flexural strength by 53% [57].

Many other fiber types have also been studied for their reinforcing effect on polymers. For most fibers, there have been increases in the composite mechanical properties. This is mainly because the high specific properties of the natural fibers normally, in the right conditions, improve the yield points of the polymer. Since the material is composite in nature, the properties of constituent materials reinforce one another. For instance, addition of 27.6 v-% okra fibers increased the tensile strength of polyester by 135% [58]. Similarly, 37.5 volume percent of the same fiber in the composite increased the Young's modulus by 81%. Similarly, reinforcement of polyester with 25 w-% coir fibers increased the tensile strength by 30% [59]. It also improved the flexural strength by 27%. In another research, it was showed that 65 weight percent of bagasse fibers was used to increase the tensile strength of polyester by 152% [52]. The flexural strength was also increased by 315%.

Related researches also showed similar trends in the reinforcing effects of natural fibers. The use of 20 w-% hemp fibers in vinyl ester increased the flexural strength of the polymer by 7% and the flexural modulus by 22.5%, while also increasing the Young's modulus by 10% [60]. Another study reported that composite reinforcement with 50 v-% bamboo fibers increased the tensile strength of polypropylene by 106%, the flexural strength by 82% and the flexural modulus by 150% [61]. The same study also showed that 15 v-% of the fiber improved the Young's modulus by 36%. Similarly, incorporation of 30 w-% abaca

fibers in polypropylene increased the tensile strength of the polymer by 45% [62]. The same weight percent of the fiber in polypropylene also the flexural strength by 35% [62]. In another study of PP composite, addition of 50 w-% rice hulls in PP increased its flexural strength by 130% [63]. The Young's modulus was also increased by 214%. The same study also showed that 50 w-% kenaf fiber in PP increases the Young's modulus by 505%.

2.4. Summary

Most plant fibers that have been studied showed the ability to reinforce polymers in a composite. This research will explore the possibility of using mormodica angustisepala for such reinforcement. It will also determine if prior chemical treatment affects the properties of the fiber and the composite. The chemicals selected were among the most commonly used in the industry and are expected to confer some changes on the fiber surface. Pre-treatments are also expected to improve the fiber-polymer adhesion and this change will be observed in the included morphological studies.

3. MATERIALS AND METHODS

3.1. Materials and their sources

The materials used include fibers, polymer and chemical surface modifiers.

The main material used is the natural fiber: Momordica Angustisepala, sourced from eastern Nigerian forest. It grows as a climbing plant, so it was cut down, thrashed, retted and washed several times, and sun-dried. Figure 3.1 shows fibers in processed form, ready for use in composite production.

Fig. 3.1. Processed fibers

The other materials used are the chemicals for surface treatment. They include:

1. Sodium hydroxide
2. Potassium hydroxide
3. Maleic anhydride
4. Methacryloxypropyl-trimethoxy**silane**
5. Dicumyl peroxide
6. Sodium hypochlorite

All chemicals were procured from VWR International Oy, Helsinki.

The polymer used for the composite is polypropylene (PP). The PP was in granular forms and was procured from Borealis Company.

3.2. Surface treatment procedures

Fiber bundles were selected from different parts of the processed fiber mass and immersed in different chemicals for surface treatment. The raw fibers were treated in the textile chemistry lab according to the procedures outlined in table 3.1 below.

Table 3.1. Fiber treatments

Chemical	Solvent	Concentrations	Times
Sodium hydroxide	Water	1%, 3%, 5% and 10% by weight	0.5hour, 3 hours and 6 hours
Potassium hydroxide	Water	1%, 3%, 5% and 10% by weight	0.5hour, 3 hours and 6 hours
Maleic anhydride	Toluene	0.5%, 1%, 3% and 5% by weight (solution placed in water bath at 70^{o}C)	0.5hour, 3 hours and 6 hours
Silane	50/50 acetone/water solution	0.5%, 1%, 3% and 5% by weight (raw fibers were first treated with 5 wt% NaOH for 1 hour, rinsed and dried for 72 hours)	0.5 hour, 1 hour and 3 hours
Dicumyl peroxide	Toluene	1%, 3%, 5% and 10% by weight (Fibers were first treated with 5 wt% NaOH for 1 hour, rinsed and dried for 72 hours)	0.5hour, 1 hour and 3 hours
Sodium hypochlorite	Water	1%, 3%, 5% and 10% by weight (Fibers were pre-treated with 5 wt% NaOH for 1 hour, rinsed and dried for 72 hours)	0.5hour, 1 hour and 3 hours

All treated fibers were rinsed with distilled water, and air dried for 72 hours. After the treatment and drying stages, all the treated fibers were packaged and labeled accordingly, and taken for observations and tests.

3.2. Fiber tests

The fibers were tested for various properties, before and after treatment. The tests were conducted, primarily to determine the basic physical and mechanical properties of the fiber, and secondarily, to observe the effect of treatment on these properties. Even though most literature has already confirmed that fiber treatment is a beneficial process in natural fiber-based polymer composite manufacture, it is still important to independently study the effect of these treatments on the individual fiber before they are incorporated in the composite. This would be helpful in understanding the factors influencing the difference between properties of polymer composites made with treated and those made with untreated natural fibers.

All tests were performed under the standard lab conditions of 20^{o}C temperature and 65% Relative humidity, RH.

3.2.1. Optical microscopy

Before proceeding to the other tests, Leica microscope was used to view some of the samples at 25x, 100x and 200x magnifications. Magnifications were obtained by manually selecting the objective lens corresponding to desired size of image. The strength and distance of the lens from the sample determined the magnification used for the observation.

This was for initial observations of the immediate effect of the treatments on the fiber, as well as to determine the diameters of selected fibers and fiber bundles. To obtain good view, adjustments were made to the focus, x- and y-axis alignment, and contrast.

3.3.2. Moisture content test

Moisture content test was performed to determine the moisture content of the fibers after the initial processing stage before the treatments. Three glass containers were weighed and recorded. Three (3) samples of fiber bundles were selected from the fiber mass and weighed. The fiber bundles were in the conditioned state (20^{o}C, 65% RH). The samples were put in the containers and subsequently placed inside an oven. The temperature of the

oven was set at 105^{o}C. The samples were left to dry inside the oven for 4 hours. After 4 hours, they were removed from the oven and placed in a desiccator. After 24 hours, the fibers and their containers were removed from the desiccator and weighed. The differences in the initial and final weights were noted.

3.3.3. Tensile test

Ten (10) specimens were selected from each fiber sample for tensile test. The diameters were measured using the optical microscope. To view the fiber, 10x lens type was used and each of its scale is equal to 10.2µm. The fibers surface were uneven, so efforts were made to categorize all the fibers into 20, 40, 60 and 80 scales corresponding to 204µm, 408µm, 612µm and 816µm respectively.

The equipment used to test for the fiber tensile properties was a Testomeric M500 mechanical testing machine. The initial length of fiber (or space in-between the jaws of the tensometer) was 20mm. Each fiber was pulled until it breaks or yields, and the force/extension plot and other data were printed from the connected computer. The speed of the test was 10mm/min and the maximum cell load was 20N. The test was performed under these conditions: 20^{o}C, 65% RH.

3.3.4. Water absorption test

Ten (10) fibers were selected from each sample and weighed to obtain the dry weight. The samples were separately immersed in distilled water and left to stand for 24 hours. They were then weighed, after 1 minute of air drying, to obtain the wet weight. The percentage water absorption of each fiber sample was then calculated thus:

[(wet weight – dry weight)/dry weight] x100 ……(1)

3.3.5. Density measurement

For each sample, the lengths of 10 fibers were measured using a measuring rule. After measurement, each fiber was weighed in a digital weighing balance to determine the mass. The length and the diameter, earlier obtained using the optical microscope, were used to compute the volume -cylindrical structure was assumed for all fibers. The density is the mass per unit volume of the fiber. Average density was calculated from the obtained data.

3.4. Polypropylene composite production

Some of the processed fibers were initially still in thick fiber bundles. These are not very suitable for composite production. Further efforts were made to separate them using the carding process. After this, more single fibers were obtained in the mix. The separation was achieved with both carding machine and later, a manual fiber shredder. Remaining long fibers were cut with a scissors.

The PP granules were mixed with the fibers in the weight ratio of 9:1 (10% fiber weight). The polymer and fibers were mixed in pans using Precisa 6000D digital weighing machine. This machine and the materials are shown in figure 3.1 below.

Fig. 3.2. Precisa 6000D digital weighing machine

After weighing, the polymers and fibers were mixed and fed into the Plasti-Coder PL2000 high shear mixer in order to melt the PP and thorough mix the composite. This process was carried out in the temperature range of 215 to 230^{o}C and mixing speed ranging from 1 to 60 revolutions per second. Several batches of composites were separately made from raw and treated fibers. Finally, some batches of plain (unreinforced) plastics were produced with only the PP –to be used for reference testing. The high shear mixer used is shown in figure 3.2 below.

Fig. 3.3. Plasti-Coder PL2000

Each batch of composite produced is taken to the MKH compression molding machine to be pressed into a flat shape. This machine uses high pressure and heat to press the soft composites into a flat shape or slab of about 4mm thickness. This slab forms inside a flat mold placed in-between the upper and lower jaws of the machine, shown in figure 3.3.

Fig. 3.4. MKH compression molding machine

The compressed materials were allowed to cool. Later, they were cut into the required shapes and dimensions for various tests.

3.5. Composites properties tests

The mechanical properties of the polymer composites were tested according to SFS/ISO (SFS-EN 15534-1) standards for testing plastics and composites [65]. The mechanical tests performed were tensile test, impact test and bending test. The ambient conditions are: 50% humidity and 18^{o}C temperature.

3.5.1. Tensile test

Tensile tests were performed on the virgin polymer, polymer reinforced with raw fibers and polymer reinforced with treated. The extensiometer speed is 5mm/min. The equipment used was Instron 5967 and was set up as shown in figure 3.4.

Fig 3.5. Tensile tester

The test pieces are dumbbell-shaped with rectangular cross-section. The test area dimensions were:

Gauge length, l_0 = 50 mm

Width, b = 10mm

Thickness, h = 4mm

3.5.2. Bending test

Bending tests were performed on the virgin polymer, polymer reinforced with raw fibers and polymer reinforced with treated fibers according to SFS/ISO standards (SFS-EN 15534-1, EN ISO 178). 3-point flexure test was adopted for the tests. The equipment used was Instron 5967 and was set up as shown in figure 3.5.

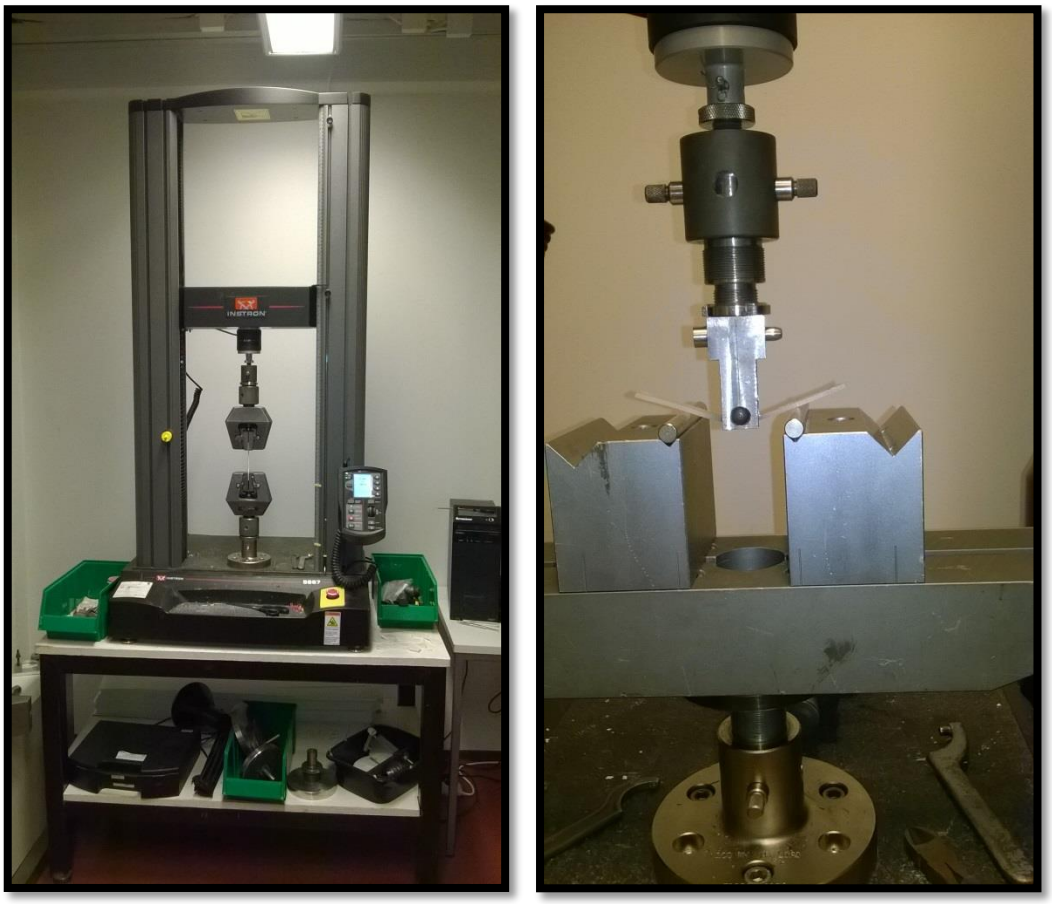

Fig 3.6. Bending tester

The test pieces are rectangular and their approximate dimensions were:

Length, l = 100 mm

Width, b = 10mm

Thickness, h = 4mm

3.5.3. Impact test

Impact tests were performed on the virgin polymer, polymer reinforced with raw fibers and polymer reinforced with treated fibers according to SFS/ISO standards (SFS-EN 15534-1, EN ISO 179-1). The equipment used was Ceast Resil 5,5. It was set up as shown in figure 3.6. The test was performed with 4J Charpy hammer; air resistance was taken into account (0.03).

Fig 3.7. Impact tester

The test pieces are rectangular and their dimensions were:

Length, l = 100 mm

Width, b = 10mm

Thickness, h = 4mm

3.5.4. Density measurements

Samples of composites were selected and the dimensions were measured using a fabric measuring guage. After measurement, each composite sample was weighed in a digital weighing balance to determine the mass. The length, width and thickness were used to compute the volume. The density is the mass per unit volume of the fiber. Average density was calculated from the obtained data.

3.5.5. Moisture content tests

Boiling test was used to study the moisture resistance of the polymer and its composites according to SFS/ISO standards (SFS-EN 15534-1, ISO-EN 317). The equipment used was Original Hanau Linitest. The test samples and the boiling machine are shown in figure 3.7 below.

Fig 3.8. Samples and boiling equipment

Specimen was weighed and immersed in boiling water at 100ºC, and then placed inside the equipment for 5 hours. The equipment contains glycol antifreeze that maintains the heating level at 100ºC, and has a rotating feature that rotates the sample holders for even heat distribution. After 5 hours, the samples were removed and excess water was quickly and gently wiped away, then the final weight was measured to determine the water uptake.

3.6. SEM procedures

The scanning electron microscope was used to obtain microscopic images of both fibers and composites. The Zeiss Ultraplus SEM was used to produce the images.

For the fibers, tiny fiber strands were extracted from the raw and treated fibers. There were 7 specimens in all, one from the raw fiber and one from each of the surface-modified fibers. The specimen were placed on the sample holders and coated with carbon to prevent

charging, a phenomenon that affects image clarity. The specimen was scanned with an acceleration voltage of 5KV and a working distance of 10mm.

Similarly, composite specimens were prepared from both composites of raw and treated fibers. The specimens were taken from around the edges that were ruptured during the tensile test. The essence was to provide a clear microscopic view of the way the fibers adhered to the polymer matrices. Fiber-matrix interface and extent of adhesion are best seen at the point of tensile-induced breakage. The specimen were placed on the sample holders and coated with carbon to prevent charging, a phenomenon that affects image clarity. The specimen was scanned with an acceleration voltage of 5KV and working distance of approximately 10mm.

4. RESULTS AND ANALYSIS

The tests performed on the fiber, polymer and composite yielded some results. The data obtained and their interpretations will be presented in this chapter.

4.1. Fiber characterization results

Results from fiber characterization provided an understanding of the properties of the fibers –raw and treated. These properties include the moisture content, density, water absorption and tensile strength. These results and morphological images of the fibers are presented in this section.

4.1.1. Visual observations

Sodium hypochlorite (NaOCl) bleached the fibers to a whitish coloration. Maleic anhydride changed the color to light gray. All other chemicals gave the fibers different shades of yellowish brown whose intensity increases with increasing concentration. Higher concentrations of maleic anhydride caused easy breakage of fibers ostensibly due to structural deformation.

Observation with optical microscope showed that there are smooth alignment of the microfibrils making up the fiber bundle for fibers treated with sodium hydroxide and silane. Such alignment could have resulted from the removal of some lignin from the fiber structure giving space for a rearrangement of the microfibrils. Other treatments only gave the fibers very minimal rearrangement. Maleic anhydride treatment produced no alignment. Images from the optical microscope are shown in figures 4.1 – 4.7 below. Better observations were made directly from the microscope than from the saved screen images. However, the extent of delignification due to treatment can slightly be discerned from the images, from the amount of free spaces inside the fiber.

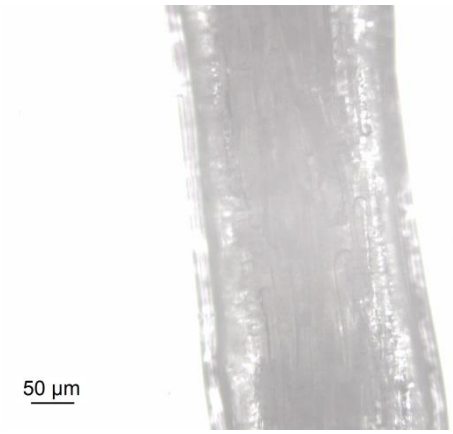

Fig. 4.1. Optical microscope image of raw fiber

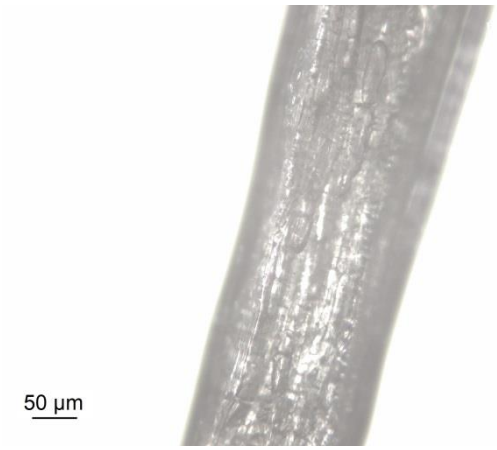

Fig. 4.2. Optical microscope image NaOH-treated fiber

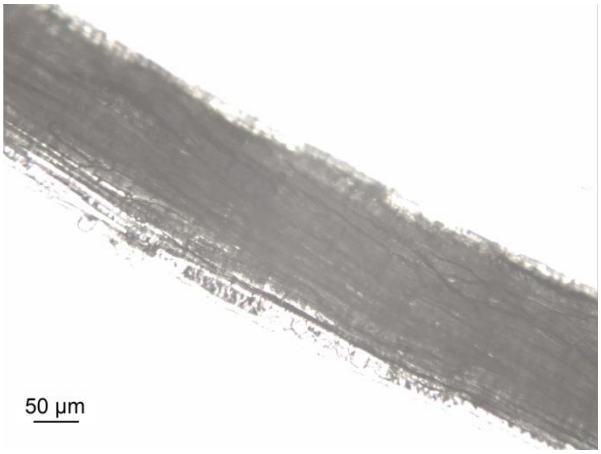

Fig. 4.3. Optical microscope image KOH-treated fiber

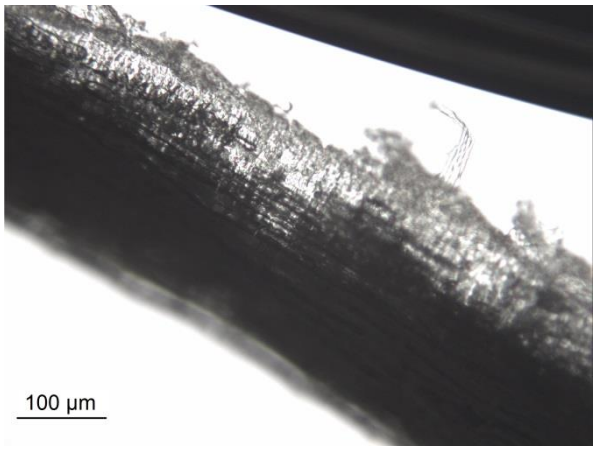

Fig. 4.4. Optical microscope image Silane-treated fiber

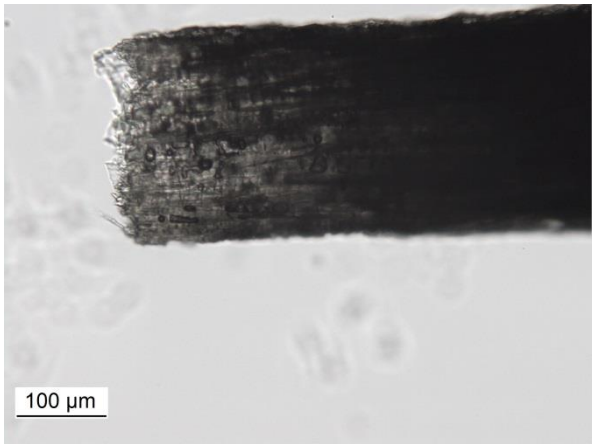

Fig. 4.5. Optical microscope image MA-treated fiber

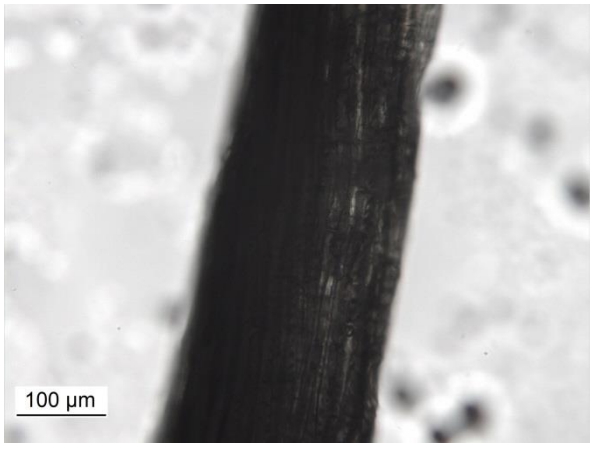

Fig. 4.6. Optical microscope image DCP-treated fiber

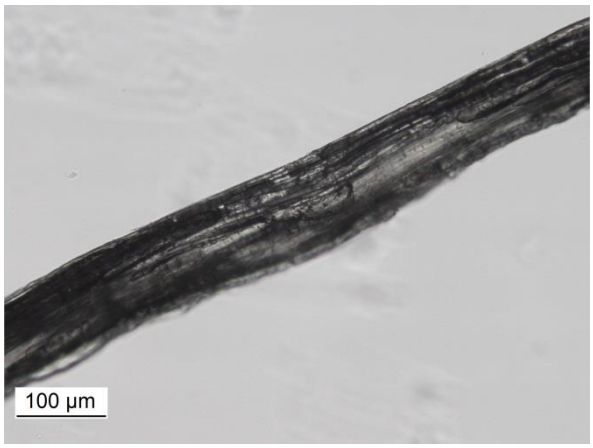

Fig. 4.3. Optical microscope image NaOCl-treated fiber

4.1.2. SEM images of fibers

Scanning electron microscopy clearly revealed the structure of the fiber bundle and the fibrils and, in some cases, showed the superficial effect on treatment chemicals on the fibers. These SEM images are shown in figures 4.8 - 4.14.

The images were taken at different magnifications of the raw and treated individual fibers, with each magnification showing the same view of the fiber and different level of details. The effect of treatment is seen to be more pronounced in NaOH-treated fiber with the occurrence of groove-like features on the surface. These features could be inferred as the side effect of the reaction between the chemical and cellulose compounds of the fiber or specifically due to the attack of the sodium ions, Na^{+}. Same features are observable on the surface of KOH-treated and silane-treated fibers, even though to a lesser degree. These features relates to reported fiber surface roughness that improves mechanical interlocking of fibers with polymer in a composite [21]. DCP-treated fiber shows some level of disfiguration of the fiber structure possibly due to the drastic action of the peroxide in contact with the fiber surface. MA-treated and NaOCl-treated fibers showed very minimal changes in the surface features. All the chemical treatments showed cleaner surfaces devoid of impurities and fatty compounds that are observable in the surface of the raw fiber.

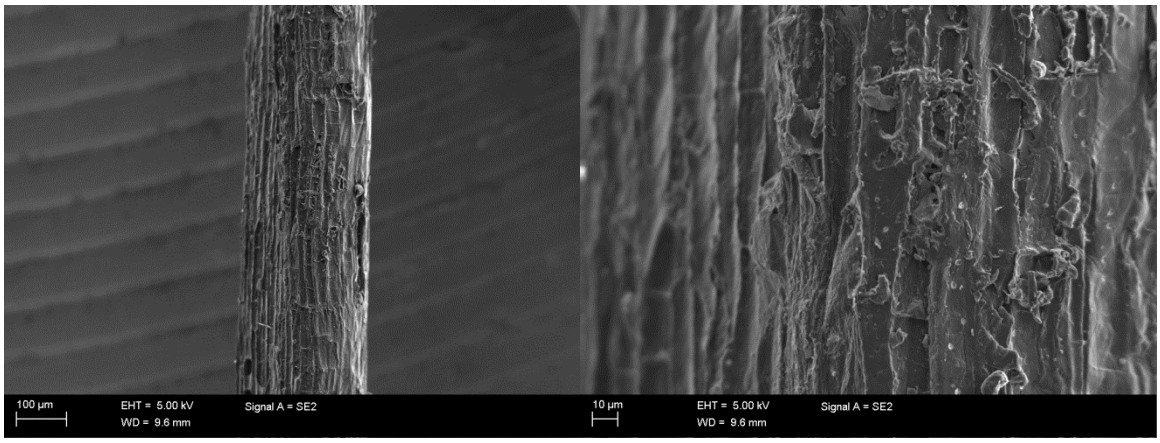

Fig. 4.8. SEM of raw fiber

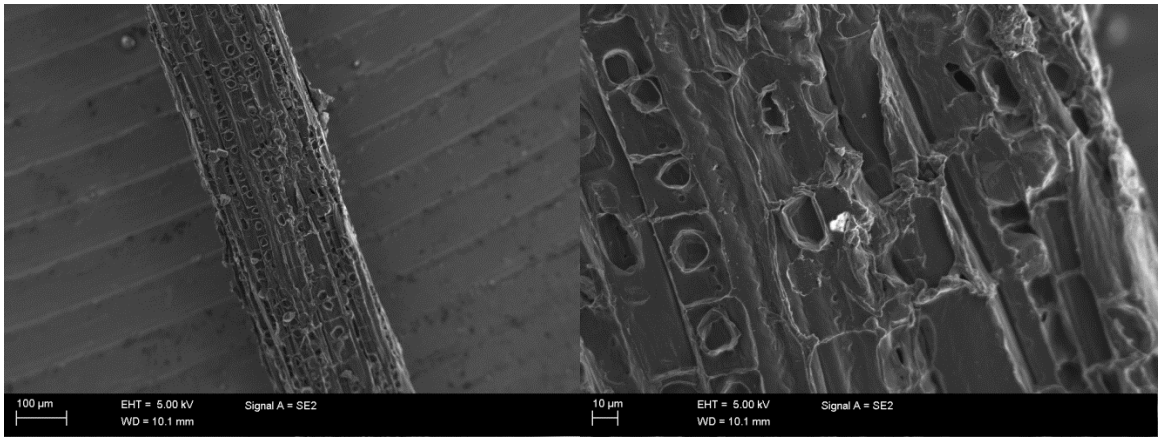

Fig. 4.9. SEM of NaOH-treated fiber

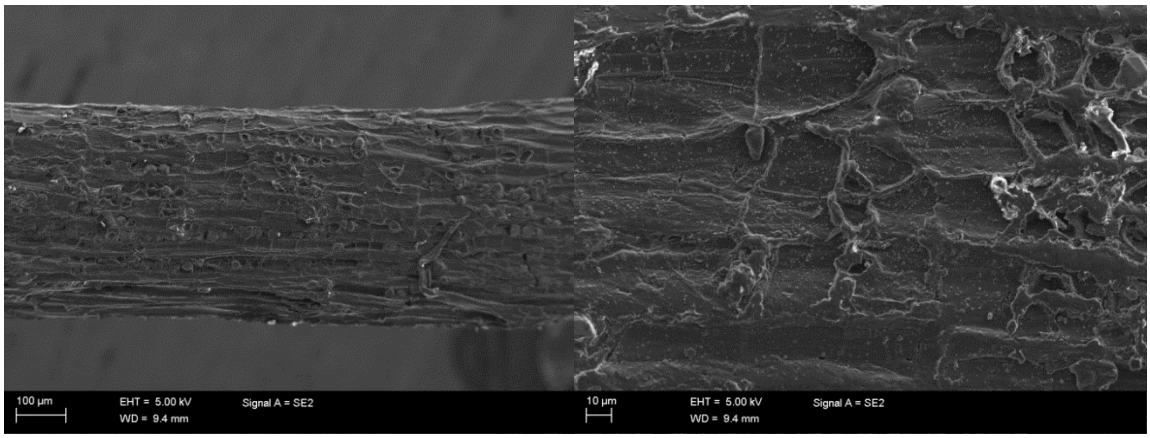

Fig. 4.10. SEM of KOH-treated fiber

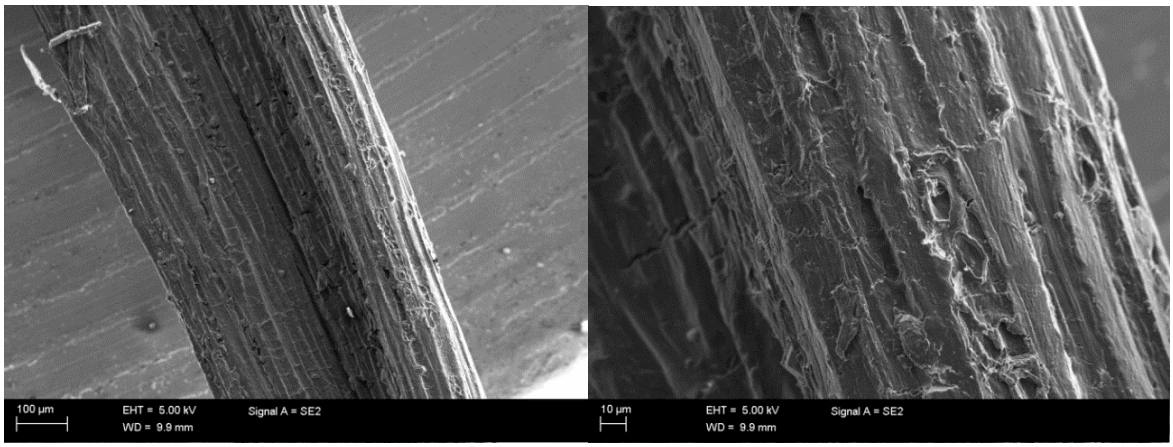

Fig. 4.11. SEM of Silane-treated fiber

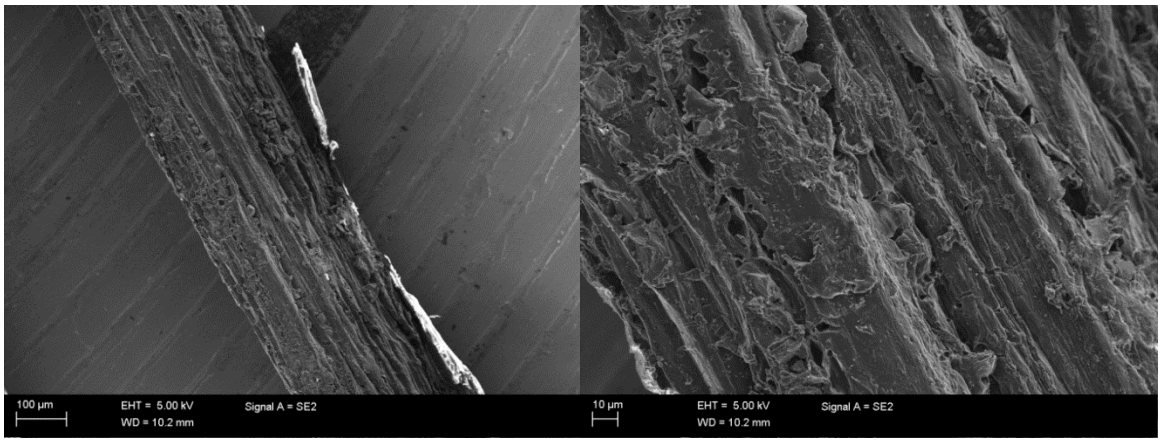

Fig. 4.12. SEM of DCP-treated fiber

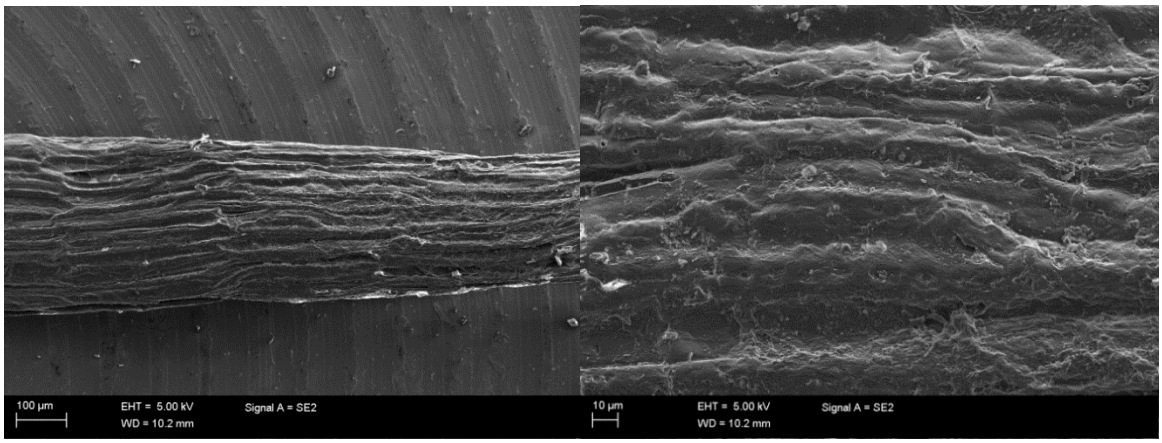

Fig. 4.13. SEM of MA-treated fiber

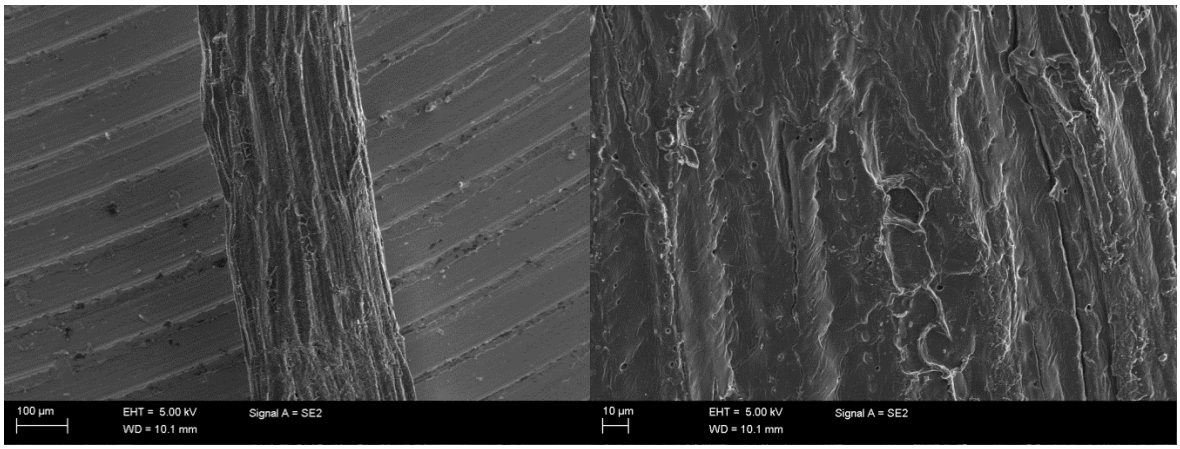

Fig. 4.14. SEM of NaOCl-treated fiber

4.1.3. Moisture content

The moisture content was determined to be 7.24%. This was obtained from the average moisture content of 3 samples after drying when compared to their initial weight at room temperature. This is presented in Table 4.1. This implies that some moisture exist in the fiber after processing from the plant sources. This is mostly from moisture absorbed from ambient environment since natural fibers are very hydrophilic. This moisture content value conforms to the normally obtained value of moisture in plant fibers [64].

Table 4.1. Moisture content of raw fiber at room temperature

	Empty glass	**Fiber before dying**	**Total before drying (g)**	**Empty glass**	**Fiber after drying**	**Total after drying (g)**	**Moisture content (g)**	**Moisture Content (%)**
Sample 1	37.04	0.8	37.84	37.04	0.74	37.78	0.06	8.11
Sample 2	44.57	0.97	45.54	44.57	0.9	45.47	0.07	7.78
Sample 3	56.54	1.09	57.63	56.54	1.03	57.57	0.06	5.83
Average					0.89		0.063	7.24

4.1.4. Water absorption

Results from fiber water absorption tests were analyzed using Minitab's Central composite design (CCD) and Response Surface Methodology (RSM). The percentage water absorption for the various concentrations and times of treatment were integrated by the software to present a clear understanding of the relationship between these parameters. The response surface and contour plots of the data are shown in figures 4.15 - 4.20.

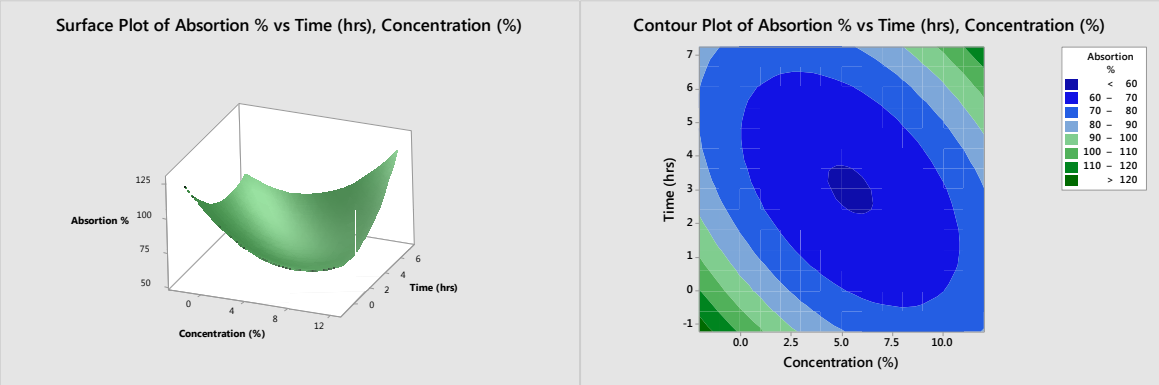

Fig. 4.15. Surface and contour plots of NaOH-treated fiber

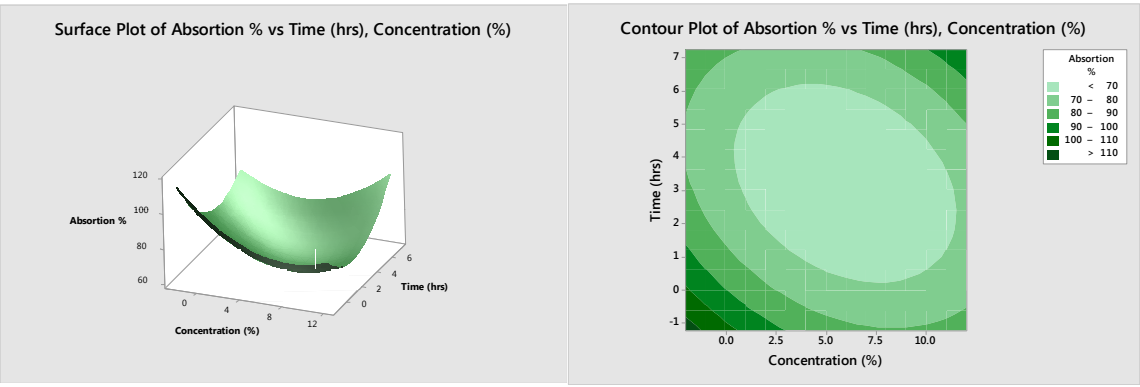

Fig. 4.16. Surface and contour plots of KOH-treated fiber

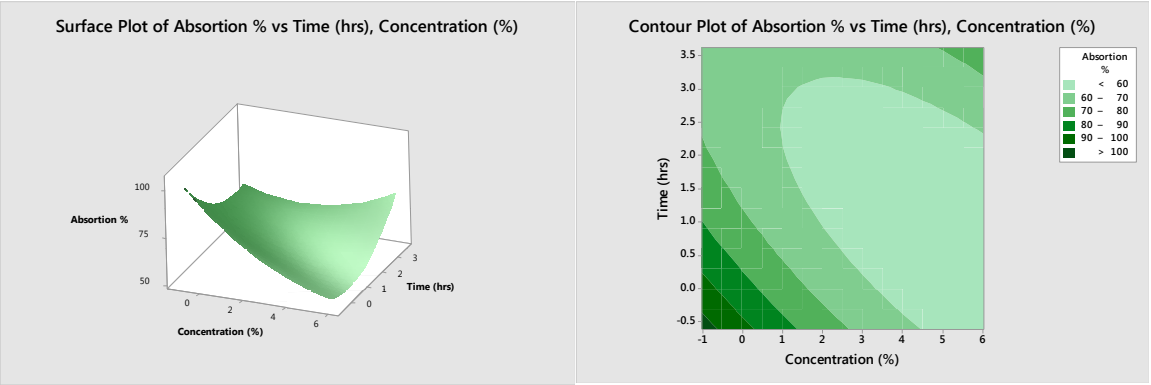

Fig. 4.17. Surface and contour plots of Silane-treated fiber

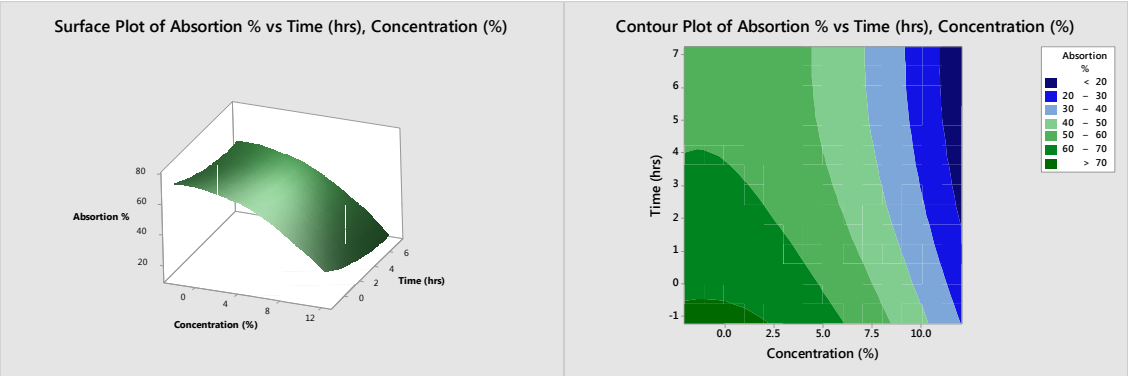

Fig. 4.18. Surface and contour plots of DCP-treated fiber

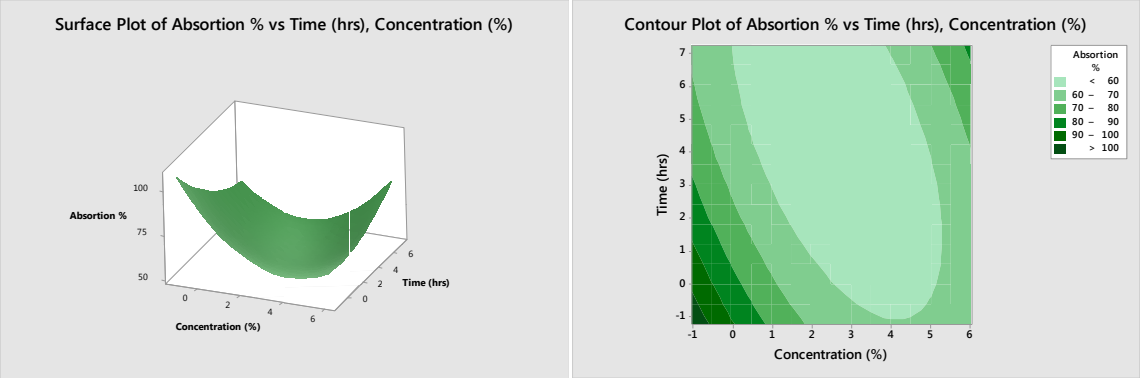

Fig. 4.19. Surface and contour plots of MA-treated fiber

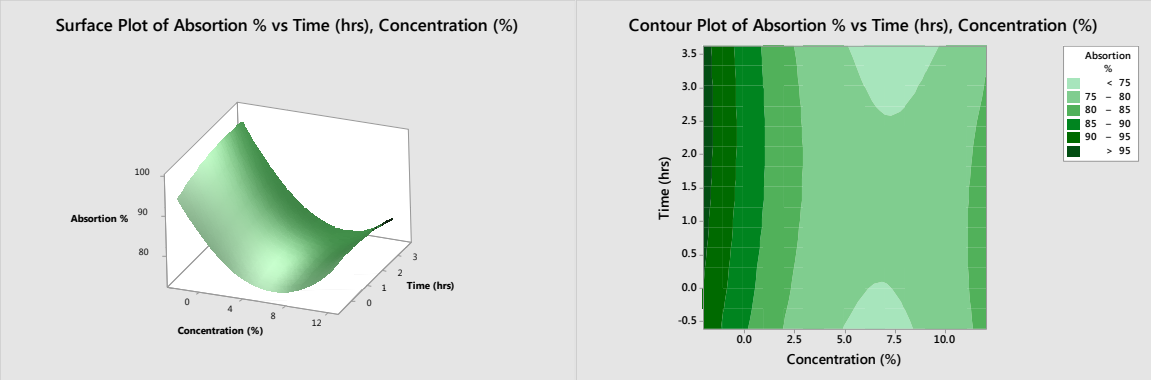

Fig. 4.20. Surface and contour plots of NaOCl-treated fiber

The surface plots represent the statistical behavior of the water absorption property at different concentrations and corresponding times. Generally the curves in the following response surface diagrams show relative decrease in percentage water absorption of the fibers with increasing concentration and time of treatment. This is the case for all types of chemical treatment used in this experiment. This reduction in the water absorption capacity of treated fibers could be attributed to the increase in hydrophobicity of these fibers due to chemical reactions with functional groups of these chemicals. These reactions usually take place at the water-attracting OH-groups of the cellulose compound and decrease their tendency to attract and absorb water molecules.

The contour plots in RSM are basically used in selecting the optimal property –in this case, water absorption- of the material. The regions in different shades of colors represent areas of similar property values, and selection is made based on the particular level or value of the property required. The percentage water absorption for raw fiber was 95%. However, percentage water absorption of 60% was achieved with NaOH, silane and MA treatments, 70% with KOH treatment, 50% with DCP treatment and 75% with NaOCl treatment. The resulting reduction in the water absorption due to the treatments is in conformity with the reported trends in water absorption property of previous surface-treated natural fibers used for composite manufacture [13-17, 25, 40].

Normally, the least percentage water absorption is desirable for fibers to be used for polymer reinforcement or composite production. But this is not considered in isolation since other properties of the fiber are also equally important, and must also be considered before making the final selection.

4.1.5. Density

Density tests were also analyzed using Minitab's Central composite design (CCD) and Response Surface Methodology. The measured density of the various concentrations and times of treatment were integrated by the software so as to easily observe the relationship between these parameters. The response surface and contour plots of the data are shown in figures 4.21 - 4.26.

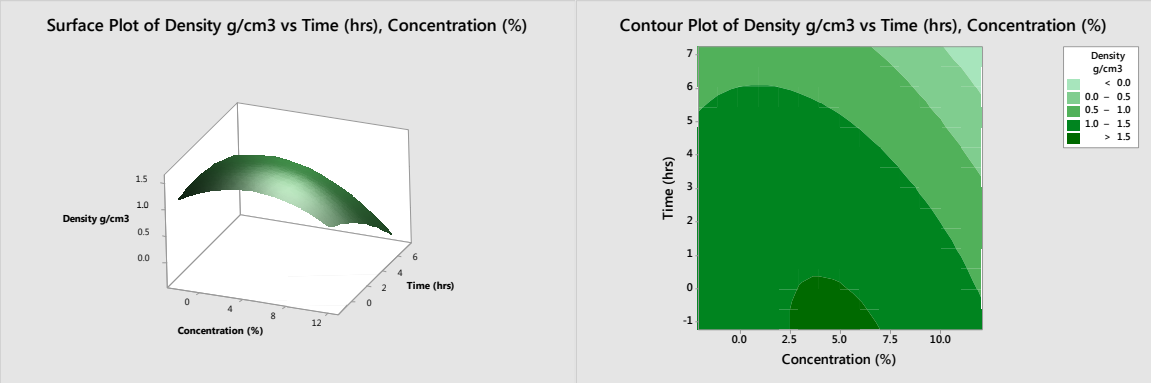

Fig. 4.21. Surface and contour plots of NaOH-treated fiber

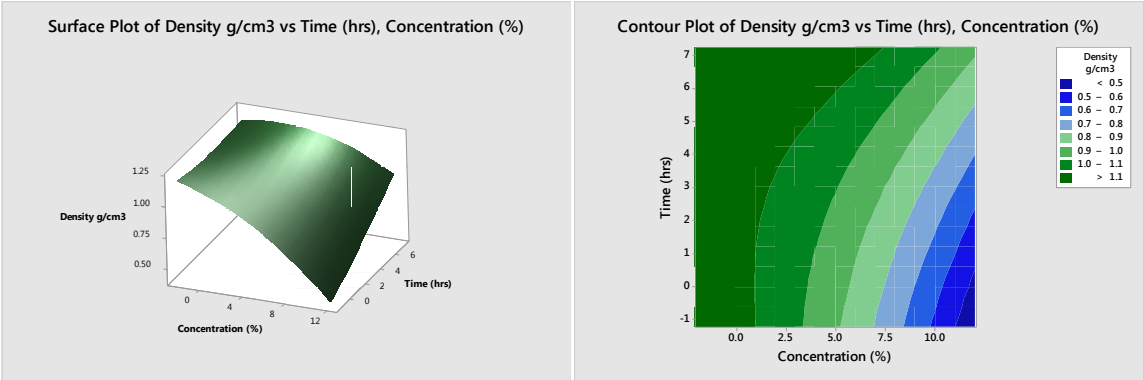

Fig. 4.22. Surface and contour plots of KOH-treated fiber

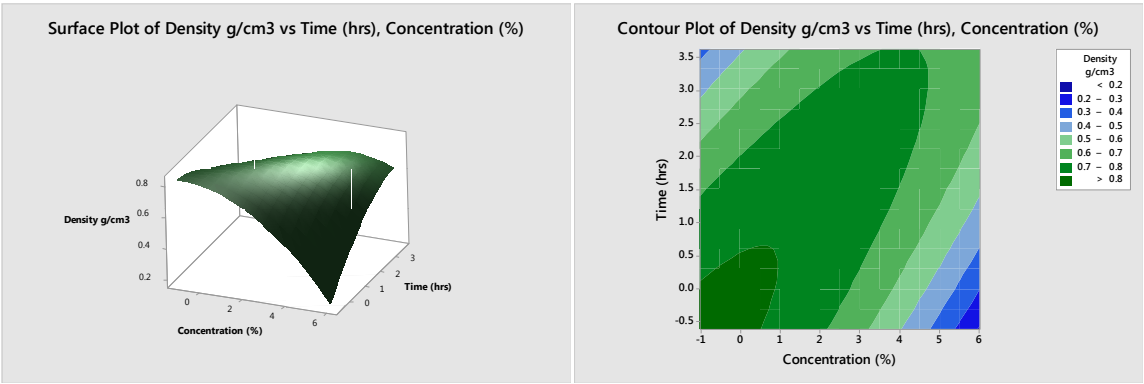

Fig. 4.23. Surface and contour plots of Silane-treated fiber

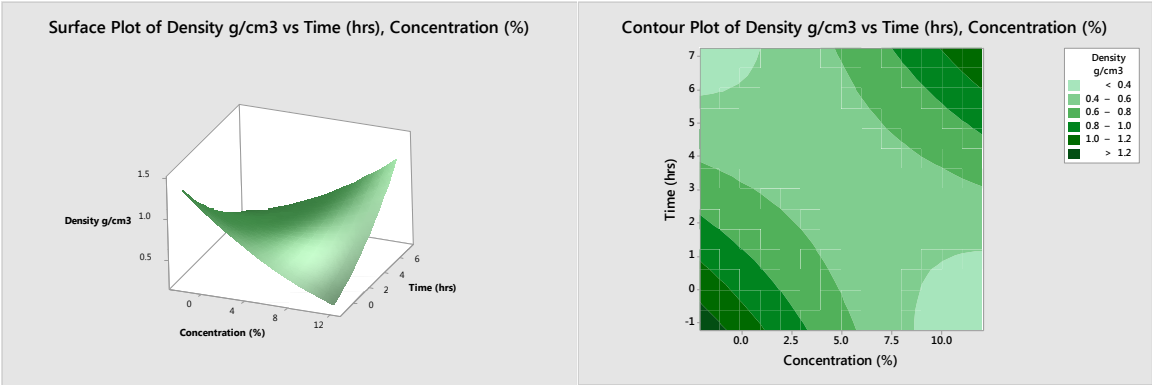

Fig. 4.24. Surface and contour plots of DCP-treated fiber

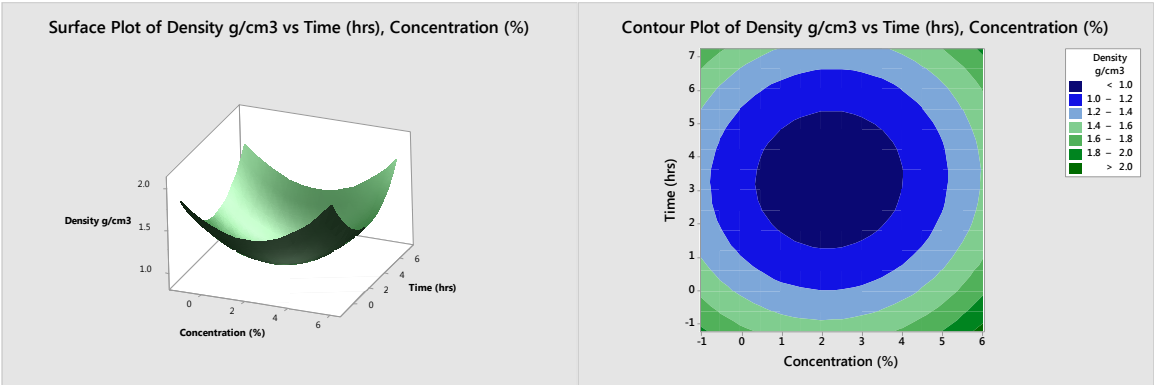

Fig. 4.25. Surface and contour plots of MA-treated fiber

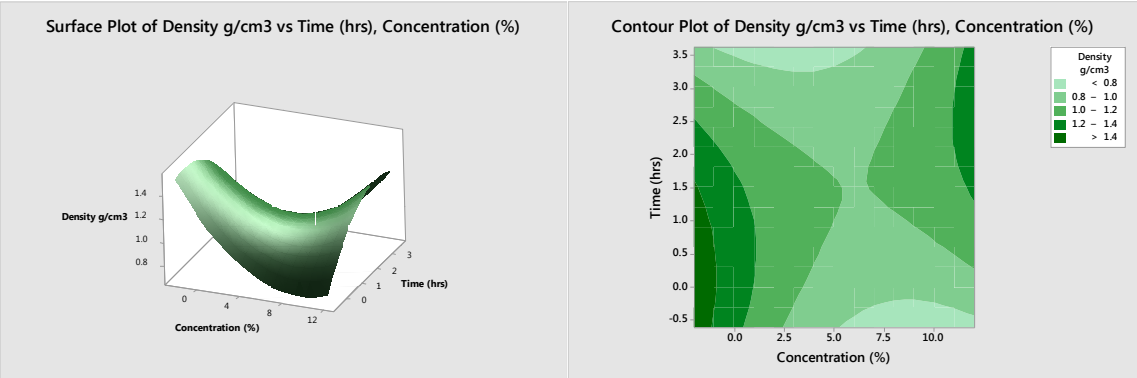

Fig. 4.26. Surface and contour plots of NaOCl-treated fiber

The surface plots show curvature that arise from the interplay of various concentrations and times of treatment. The peak of each curve represents the point or the particular concentration and time that offers the best value of density. This is more clearly represented in the contour plots which constitute different delineated regions showing different property ranges. The best density can easily be seen and selected from these regions.

Again, the contour plots were used to select the optimal density of the material. The regions in different shades of colors represent areas of similar property values, and selection is made based on the particular level or value of the property required. The bulk density of raw fiber was 1.11g/cm^3. However, an average density of 1.0g/cm^3 was achieved with NaOH, KOH and NaOCl treatments, 0.6 g/cm^3 with silane and DCP treatments, and 1.1g/cm^3 with MA treatment. On the overall, there were reductions in the density of the fibers due to the treatments. This invariably implies removal of considerable amounts of lignin and other contents from the fiber mass by the chemical treatments [18].

Density gives an idea of the amount of lignin and other non-fibrous content still remaining in the fiber bundle after the treatment. A high density will therefore imply a less effective treatment. For composite manufacture, light density may be preferable but this is dependent on the strength of such light fibers since low density could sometimes be due to fiber degeneration from excessive chemical reactions occurring at high concentration and times. Hence, selection for best density, just as for water absorption, is made while also considering the tensile strength and modulus of the treated fibers.

4.1.6. Tensile strength

Tensile tests yielded results that were analyzed using Minitab's Central composite design (CCD) and Response Surface Methodology. The software integrated the tensile strength for the various concentrations and times of treatment and provided values and plots depict-

ing the relationship between these parameters. The response surface and contour plots of the data are shown in figures 4.27 - 4.32.

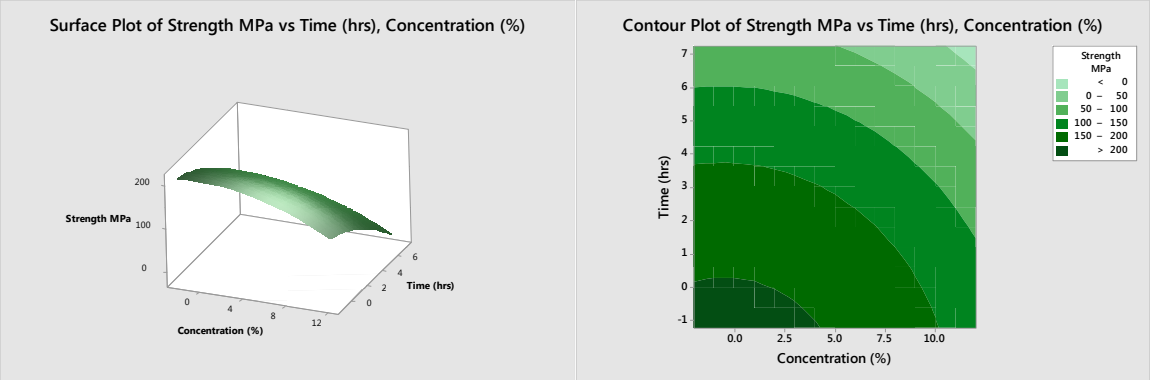

Fig. 4.27. Surface and contour plots of NaOH-treated fiber

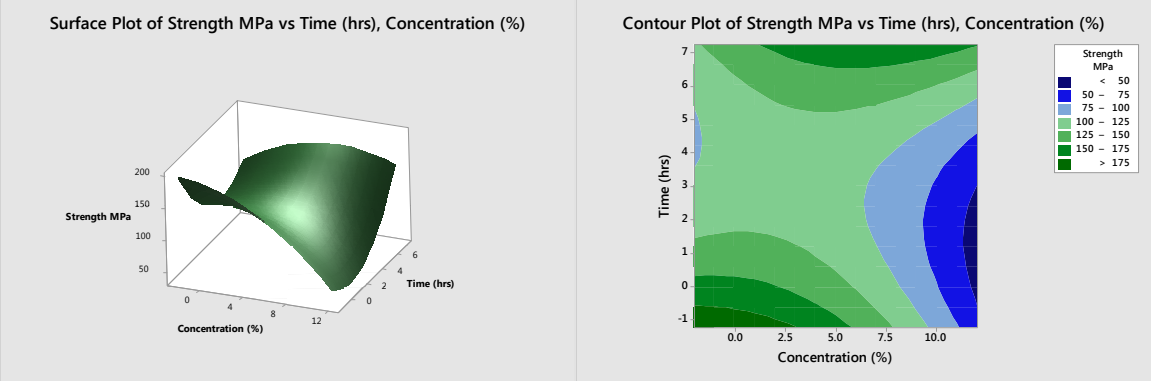

Fig. 4.28. Surface and contour plots of KOH-treated fiber

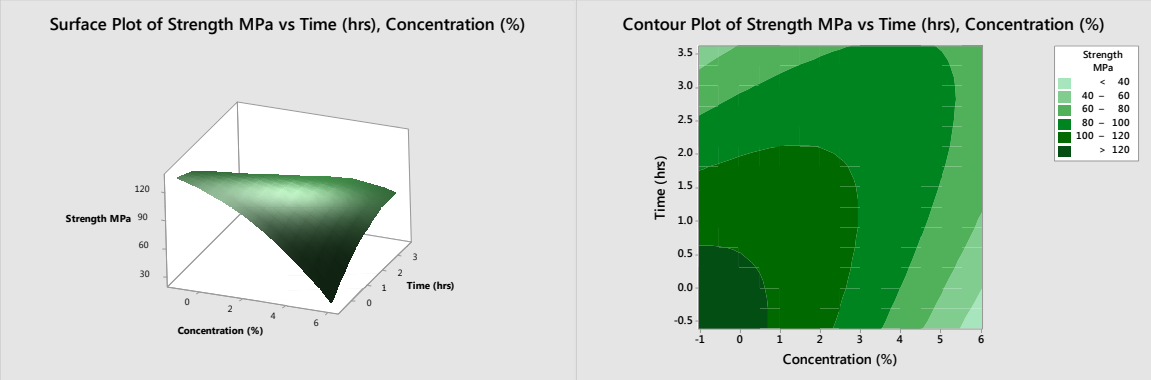

Fig. 4.29. Surface and contour plots of Silane-treated fiber

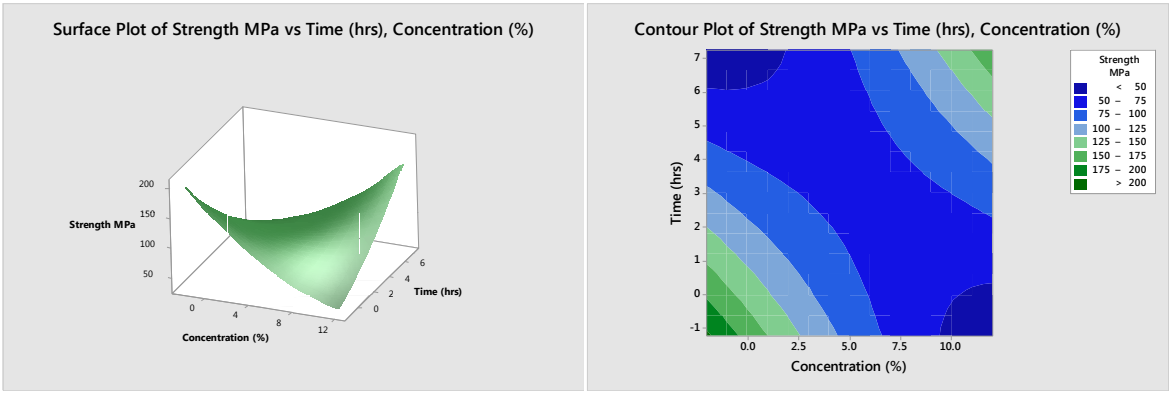

Fig. 4.30. Surface and contour plots of DCP-treated fiber

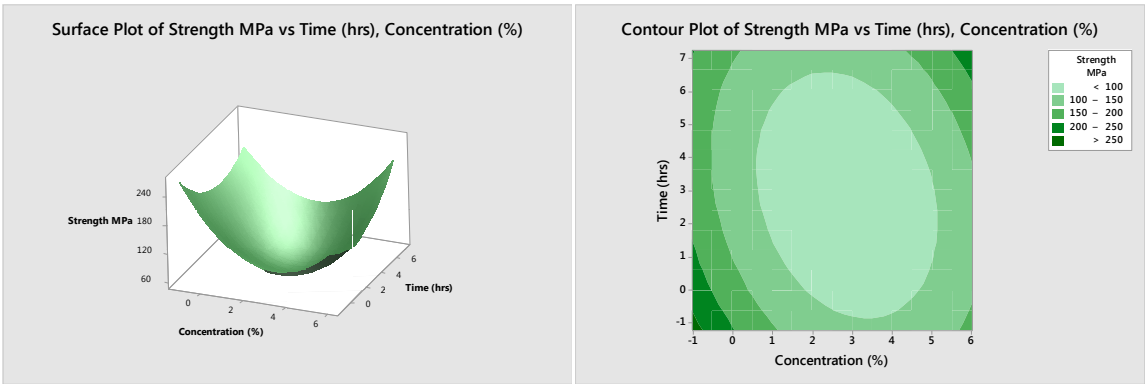

Fig. 4.31. Surface and contour plots of MA-treated fiber

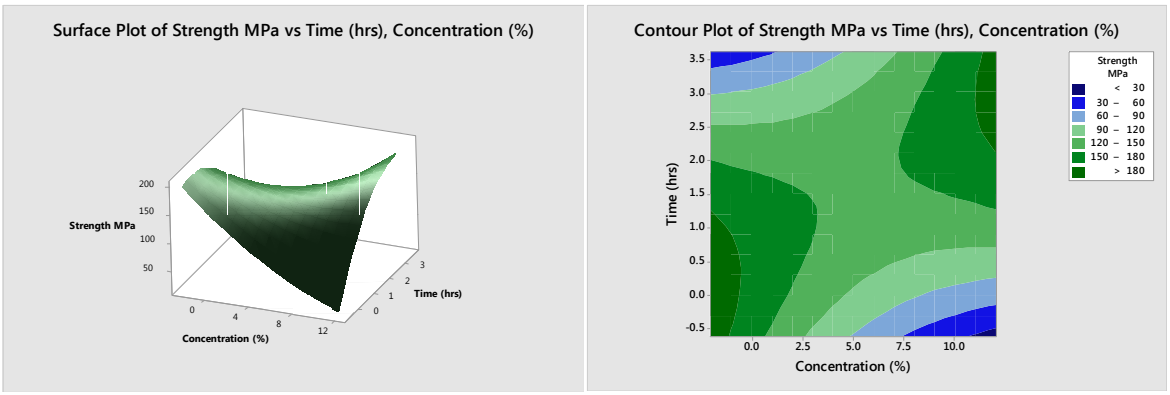

Fig. 4.32. Surface and contour plots of NaOCl-treated fiber

The peaks of the curve of the response surfaces show the maximum strength values while in the contour plots, concentrations and times with close strength values are demarcated. The best strength for composite manufacture is usually the maximum strength, hence the concentration and time of treatment that yielded the highest tensile strength is considered optimal. This will be considered in more detail in the fiber selection section.

The optimal property value was picked from the contour plots. Optimal strength of 200MPa was achieved with NaOH, 180MPa with NaOCl treatments, 150MPa with KOH and MA treatments, 125MPa with DCP treatment, and 120MPa with silane treatment. There were wide variations in the measured strength of fibers with the different times and concentration of chemicals. This is because, no two natural fibers were exactly the same

due to possible differences in structure and also differences in growing environment and conditions [9]. By and large, the property values helped to indicate the type of treatment that ensured that the fibers retain the highest amount of strength after the treatment. The obtained values of tensile strength is in concordance with reported values for similar plant fibers [9].

4.1.7. Tensile modulus

Tensile tests also provided results on Young's modulus of fibers; these results were also analyzed using Minitab's Central composite design (CCD) and Response Surface Methodology. The software integrated the Young's modulus data for the various concentrations and times of treatment and provided values and plots depicting the relationship between these parameters. The response surface and contour plots of the data are shown in figures 4.33 - 4.38.

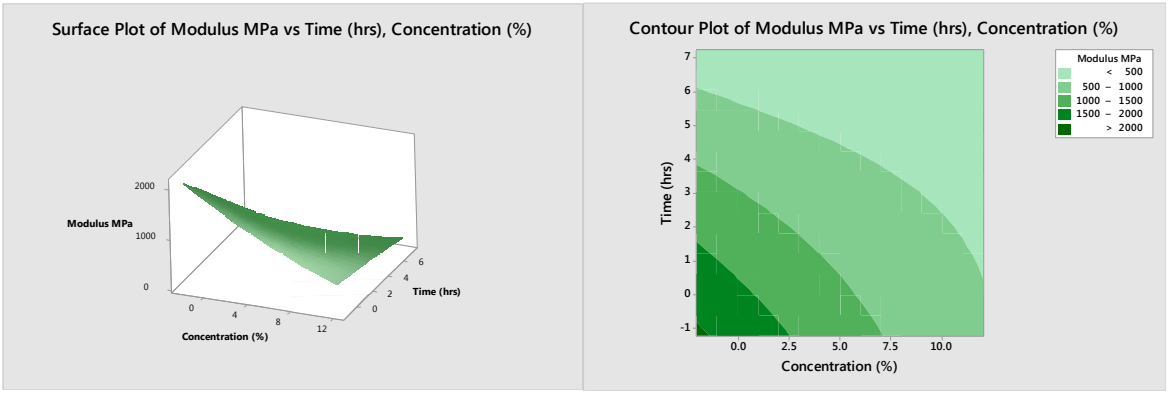

Fig. 4.33. Surface and contour plots of NaOH-treated fiber

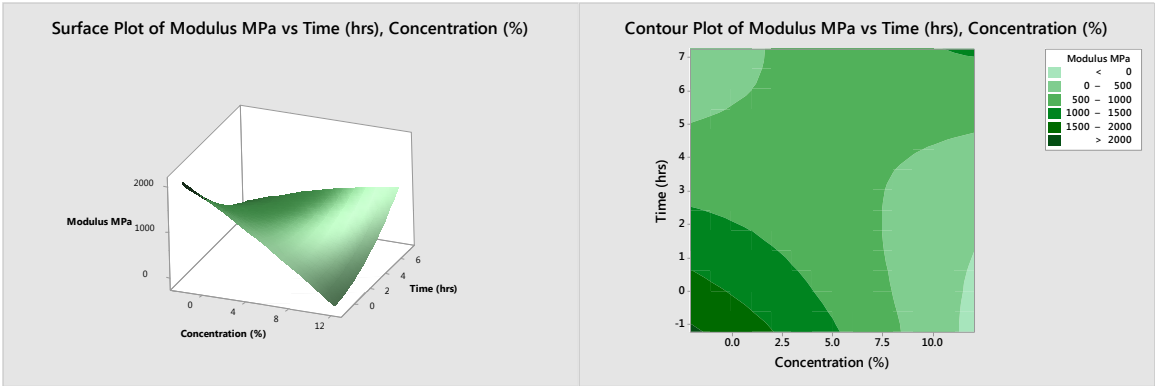

Fig. 4.34. Surface and contour plots of KOH-treated fiber

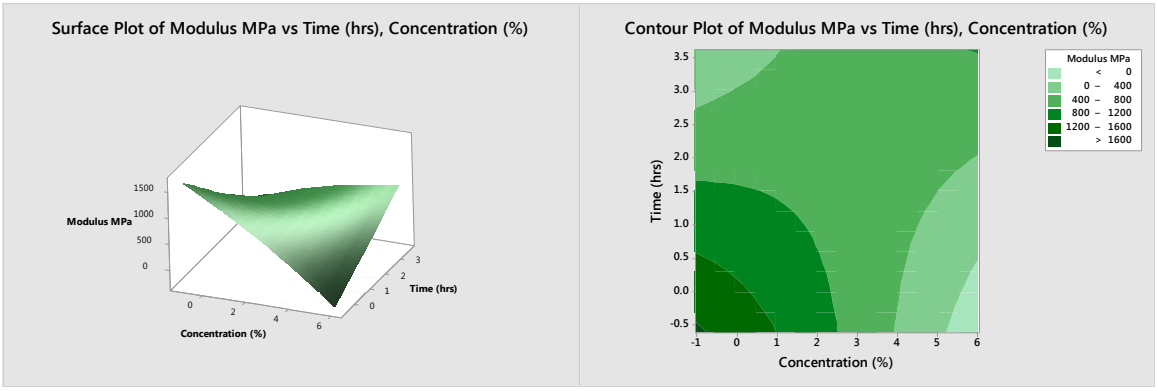

Fig. 4.35. Surface and contour plots of Silane-treated fiber

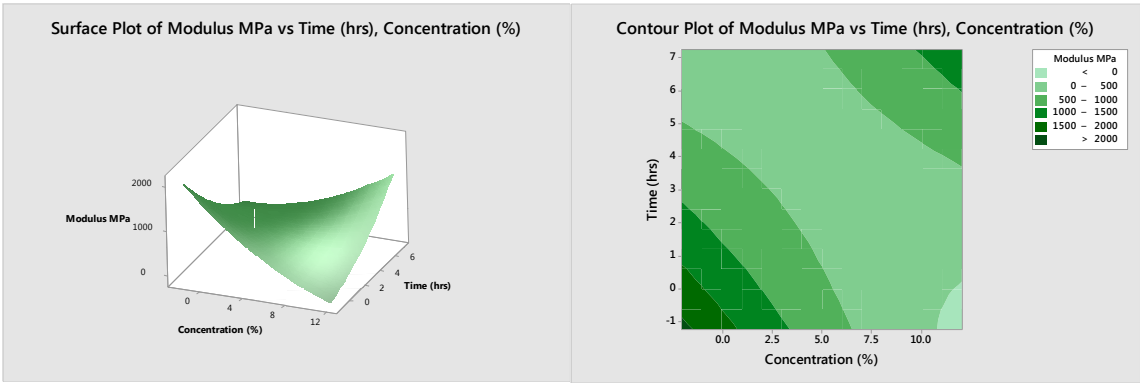

Fig. 4.36. Surface and contour plots of DCP-treated fiber

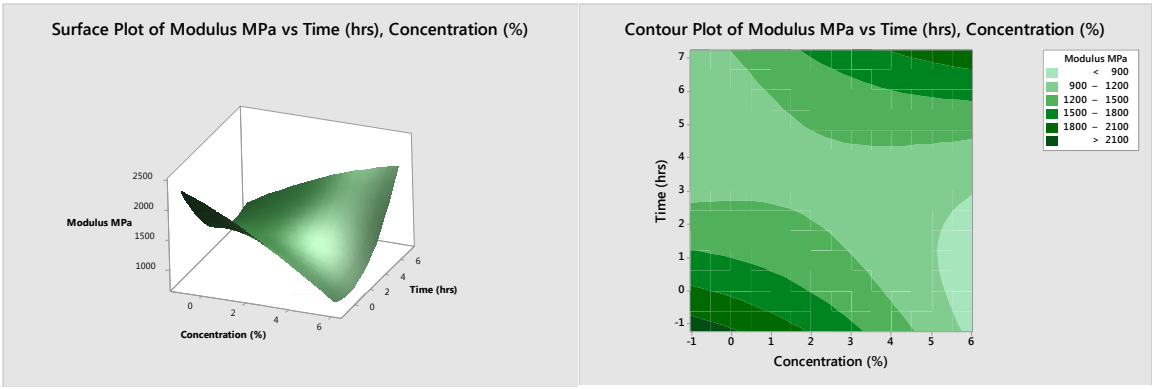

Fig. 4.37. Surface and contour plots of MA-treated fiber

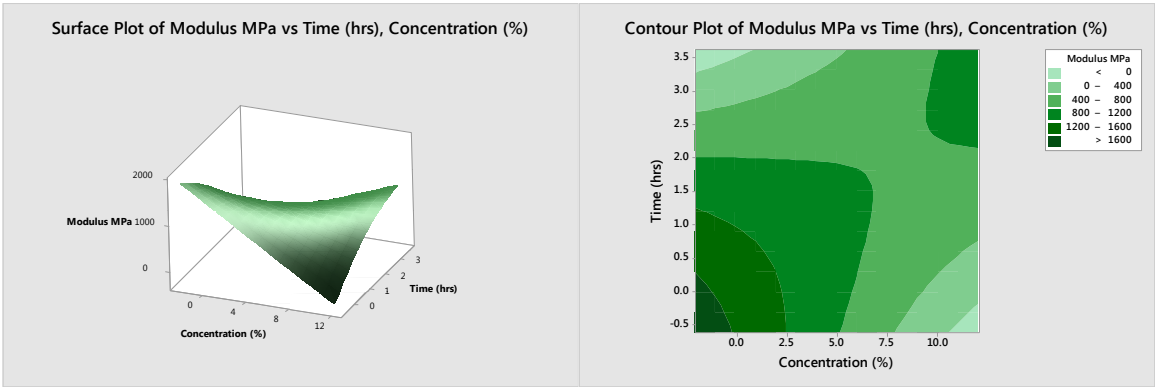

Fig. 4.38. Surface and contour plots of NaOCl-treated fiber

The plots show the property (modulus) changes of fibers tested at different concentrations and times. The response surface plot peaks at the combination or setting that yields the maximum modulus. This is different for different treatments. Just as for tensile strength, maximum modulus is desirable for fibers to be used in composite production since high modulus implies high toughness.

Here, the optimal property value was also picked from the contour plots. Optimal Young's modulus of 1500MPa was achieved with NaOH, MA and NaOCl treatments, while 1000MPa was achieved with KOH, silane and DCP treatments. Just as in strength values, wide variations were also recorded in the modulus values of the tested fibers, at the different times and concentration of chemicals. The values obtained for Young's modulus of the fibers correspond with some reported values for similar plant fibers, e.g. coconut fibers [9].

The highest concentration and time of treatment did not always translate to the highest Young's modulus because the fiber bundle weakened at such high settings, ostensibly due to excess lignin removal [25]. The higher the modulus, the more suitable the fiber is for composite production. However, all properties would be considered simultaneously in the next section to select the best setting for fiber treatment used for the composite production.

4.1.8. Fiber selection

The selection is made based on the consideration for the settings that yielded the best fiber tensile strength and modulus after treatment. This is the case because strength consideration is paramount for the intended application: composites. Other properties are equally important, but it has been observed that any level of chemical treatment improves hydrophobicity of the fibers and decreases the density too. In most cases, the higher the concentration and time setting, the better the treatment, which implies that the highest concentration and time setting often yield the lowest water absorption and density values (but not the best strength and modulus). Hence it is advisable to make selections from the optimal tensile properties.

Best settings can be observed from the contour plots, and also from the response surface. Often, the settings fall within a range of concentration, and time. It is therefore pertinent to select one feasible from the range.

Table 4.2. Best fiber treatments

Treatment	Best	Remarks
NaOH	5% concentration, 1hour time =200MPa	This setting also yields good modulus =1500MPa
KOH	5% concentration, 1hours time =150MPa	Also yields an optimal modulus =1000MPa
Silane	1% concentration, 0.5hours time =120MPa	Also yields an optimal modulus =1000MPa
DCP	1% concentration, 1hours time =125MPa	Also yields an optimal modulus =1000MPa
Maleic Anhydride	1% concentration, 1hours time =150MPa	Also yields an optimal modulus =1500MPa
NaOCl	1% concentration, 0.5hour time =180MPa	Also yields an optimal modulus =1500MPa

Based on the above selections, NaOH 5%, 1 hour was used for the treatment of fibers needed for composite production, since the setting produces the strongest fibers. It also quite easy to use on industrial scale since its solvent is water. It is also economically expedient to note that sodium hydroxide is one of the least expensive of the treatment chemicals, and more so at such low concentration.

4.2. Mechanical properties of polymer and composites

4.2.1. Tensile strength

The tensile strength values and percentage changes occurring as a result of reinforcement are given in table 4.3. The percentage change indicates the effect of the fiber on the tensile strength of the original polymer, and also the effect of pretreatment of the fiber on the property of the composite.

Table 4.3. Tensile strength of materials

Material	Tensile strength (MPa)	Percentage change
Polypropylene	28.49	
PP + raw fiber	18.79	34.05% decrease
PP + treated fiber	21.57	14.4% increase from PP+Raw fiber 24.29% decrease from normal PP

The tensile strength of the polymer decreases on addition of raw fibers. This low tensile strength was improved by the pretreatment of fibers as can be seen from the above table. Figure 4.39 is a bar chart of the tensile strength of the respective materials.

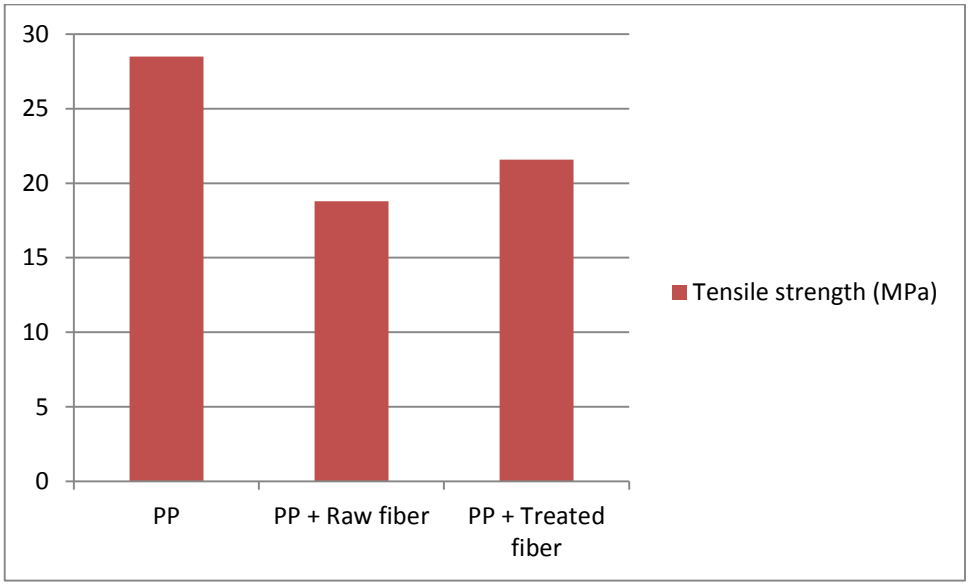

Fig 4.39. Comparison of tensile strength of materials

The tensile strength of the composite was increased by 14.4% due to the chemical treatment which induced greater adhesion between the polymer matrix and the fibers. The lower strength value of the composite compared to that of the polymer could be due to insufficient fiber component in the mixture. It could also be because the discontinuity of the short fibers created much irregularity in the polymer structure and hence weakened the frame. Longer fibers or higher volumes could offer high strength.

The increase in strength of composite due to the chemical treatment conforms with the reported trends in the industry where fiber surface treatment have been reported to improve

the tensile strength of polymer composites reinforced with natural fibers [22, 36, 37]. The alkali treatment improved mechanical interlocking of the fibers in the matrix while also inducing a chemical linkage between the cellulose and polymer chain [21]. These phenomena improved adhesive bonding of the materials and invariably, the strength of the composite.

4.2.2. Tensile modulus

The tensile or Young's modulus values and percentage changes due to reinforcement are given in table 4.4. The percentage change indicates the effect of the fiber on the tensile modulus of the original polymer, and also the effect of pretreatment of the fiber on this property of the composite.

Table 4.4. Young's modulus of materials

Material	Young's modulus (MPa)	Percentage change
Polypropylene	1093.12	
PP + raw fiber	1080.63	1.14% decrease
PP + treated fiber	1115.37	3.22% increase from PP+Raw fiber 2.06% increase from normal PP

Figure 4.40 is a bar chart of the Young's modulus of the respective materials. The Young's modulus of polymer is slightly higher than that of the raw fiber composite but lower than that of the treated fiber composite.

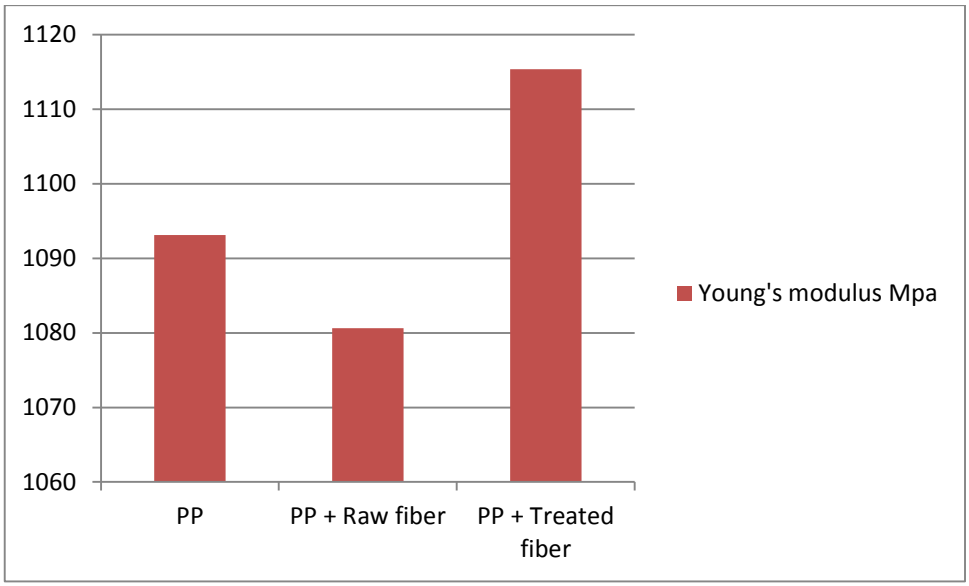

Fig 4.40. Comparison of Young's modulus of materials

Chemical treatment of the fibers resulted in 3.2% increase in elastic modulus of the composite and 2% increase when compared to the modulus of the polymer. This increase in modulus of the material could be attributed to the increase in stiffness of the polymer when fibers were added. The treated fiber composite felt "woodier" than the polymer as a result of stronger bonding of the fiber and the polymer, and this apparently affected the elasticity of the polymer.

The increase in Young's modulus of the composite arising from fiber reinforcement is normal and had been severally reported by researchers [49, 56, 61]. The fibers ostensibly made the polymer stiffer than before and increased the stress level required to induce a given amount of strain on the composite. More fiber content could further increase this feature and then increase modulus of the material. Chemical treatment caused greater adhesion between fibers and polymer and hence, more improvement in the property.

4.2.3. Elastic strain

The values of elongation at maximum load and percentage changes due to reinforcement are given in table 4.5. The percentage change indicates the effect of the fiber on the elasticity of the original polymer, and also the effect of pretreatment of the fiber on this property of the composite.

Table 4.5. Strain at maximum load of materials

Material	Strain (mm/mm)	Percentage change
Polypropylene	0.08	
PP + raw fiber	0.05	37.5% decrease
PP + treated fiber	0.05	No change from PP+Raw fiber 37.5% decrease from normal PP

Figure 4.41 is a bar chart of the strain at maximum load of the respective materials. Addition of fibers to the polymer reduces the strain on it.

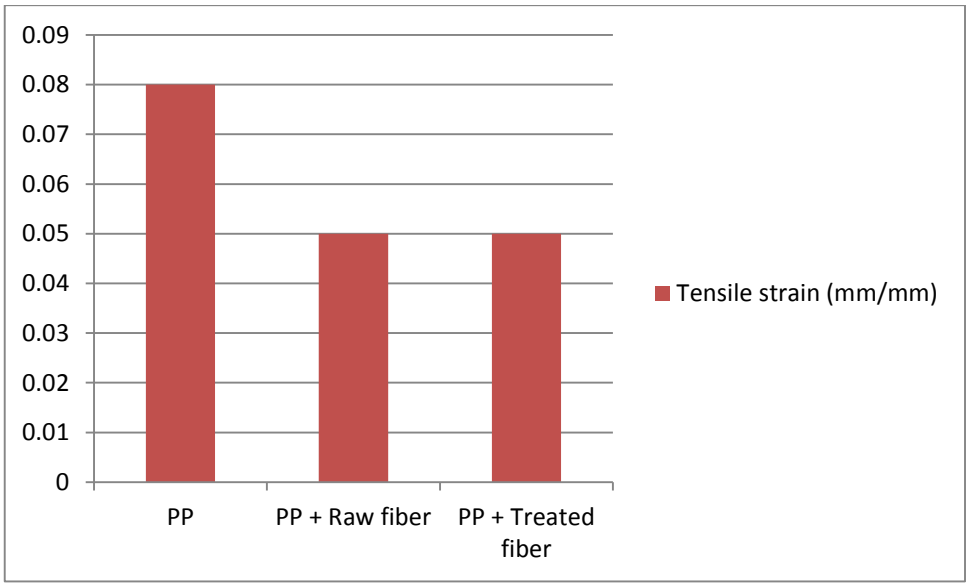

Fig 4.41. Comparison of strain at maximum load of materials

The presence of the fibers in the matrix constrains the stretching of the matrix and reduces its elasticity. This lowers the extent of strain occurring within the polymer during loading. Fiber treatment obviously has no effect on this trend as can be seen from the results in the above table 4.5.

Both raw- and treated-fiber composite show 37.5% decrease in strain when compared with that of the polymer. This property is usually not studied in composite engineering. Either low or high strain could be desirable depending on the application. For a constant amount

of stress on a material, high strain value would imply low Young's modulus and vice versa.

4.2.4. Bending strength

The bending or flexural strength values and percentage changes occurring as a result of reinforcement are given in table 4.6. The percentage change indicates the effect of the fiber on the flexural strength of the original polymer, and also the effect of pretreatment of the fiber on this property of the composite.

Table 4.6 . Flexural strength of materials

Material	Flexural strength (MPa)	Percentage change
Polypropylene	38.89	
PP + raw fiber	37.88	2.61% decrease
PP + treated fiber	36.19	4.46% decrease from PP+Raw fiber 6.95% decrease from normal PP

The flexural strength of a material represents the highest applicable stress in the material at the point of rupture or bending failure. Figure 4.42 is a bar chart of the flexural strength of the respective materials.

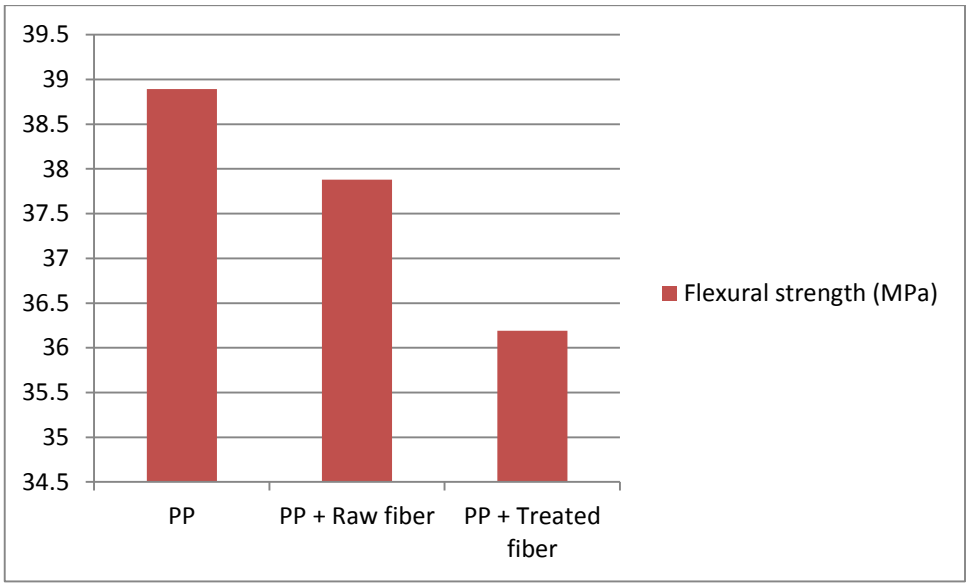

Fig 4.42. Comparison of flexural strength of materials

The flexural strength of PP was reduced by 2.61% on the introduction of raw fibers, and by 6.95% on the introduction of treated fibers. Hence it could be inferred that incorporation of small volume of short fibers in the thermoplastic polymer weakens the capacity of the polymer to withstand bending-induced stress or flexural stress.

This decrease in the flexural strength of polymer after the addition of fibers negates the report of previous researches on the same subject [49-52, 55, 57, 59, 62]. However, it is noteworthy that higher weight and volume fractions were employed in these researches. Also variations in fiber, type, source and composite processing methods employed could have contributed in the discrepancies in the results.

4.2.5. Bending modulus

The bending or flexural modulus values and percentage changes occurring as a result of reinforcement are given in table 4.7. The percentage change indicates the effect of the fiber on the flexural modulus of the original polymer, and also the effect of pretreatment of the fiber on the flexural modulus of the composite.

Table 4.7. Flexural modulus of materials

Material	Flexural modulus (MPa)	Percentage change
Polypropylene	1820.4	
PP + raw fiber	1800.15	1.11% decrease
PP + treated fiber	1480.82	17.74% decrease from PP+Raw fiber 18.65% decrease from normal PP

Flexural modulus represents the magnitude of resistance a material exhibits against bending. It is a measure of the extent of bending load a material can support when used in a component. Figure 4.43 is a bar chart of the flexural modulus of the respective materials.

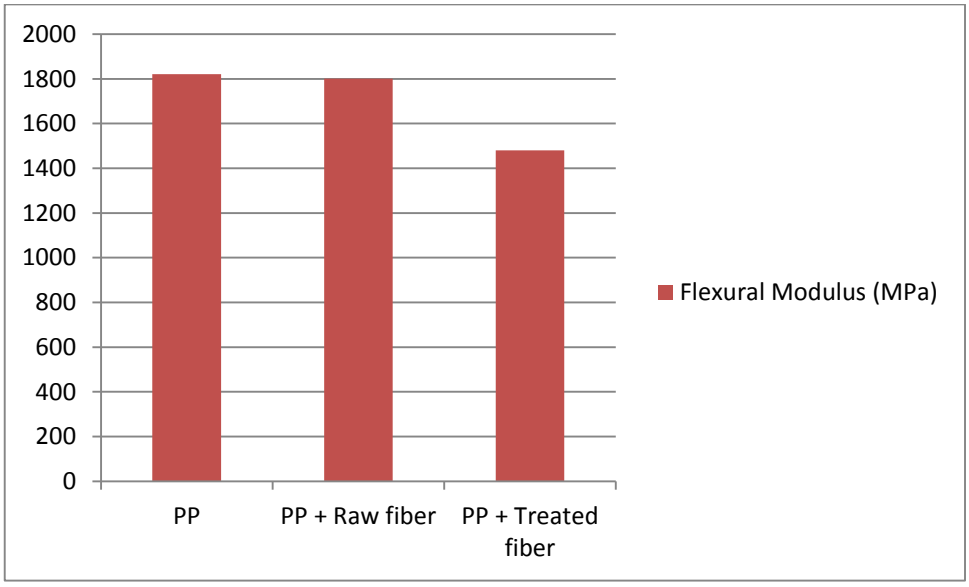

Fig. 4.43. Comparison of flexural modulus of materials

The polymer shows a minimal change in flexural modulus when raw fibers were used in forming a composite. Treated-fiber composite, however, shows a decrease of 18.65% from the flexural modulus of the polymer and 17.74% decrease from that of the raw-fiber composite. Hence, a homogenous PP could support bending loads much better than PP composites produced with 10%-wt. short mormodica angustisepala fiber. Apparently, the fibers create crack initiation sites and these accelerate the deformation process under when then material is subjected to a bending load. Fiber pretreatment seems to exacerbate this trend with the increased adhesion which increases the tearing action or bending-induced shear

force within the layers of the matrix. It would seem that this property increases with increase in weight percent fiber [49, 56, 61].

4.2.6. Impact strength

The values of the impact strength and percentage changes due to reinforcement are given in table 4.8. The percentage change indicates the effect of the fiber on the impact strength of the original polymer, and also the effect of pretreatment of the fiber on this property of the composite.

Table 4.8. Impact strength of materials

Material	Impact (J/mm^2)	Percentage change
Polypropylene	62.593	
PP + raw fiber	24.143	61.43% decrease
PP + treated fiber	21.337	11.62% decrease from PP+raw fiber 65.91% decrease from normal PP

The impact strength of the polymer decreased on addition of the fibers, both in the raw- and treated-fiber composites. Figure 4.44 is a bar chart of the impact strength of the respective materials.

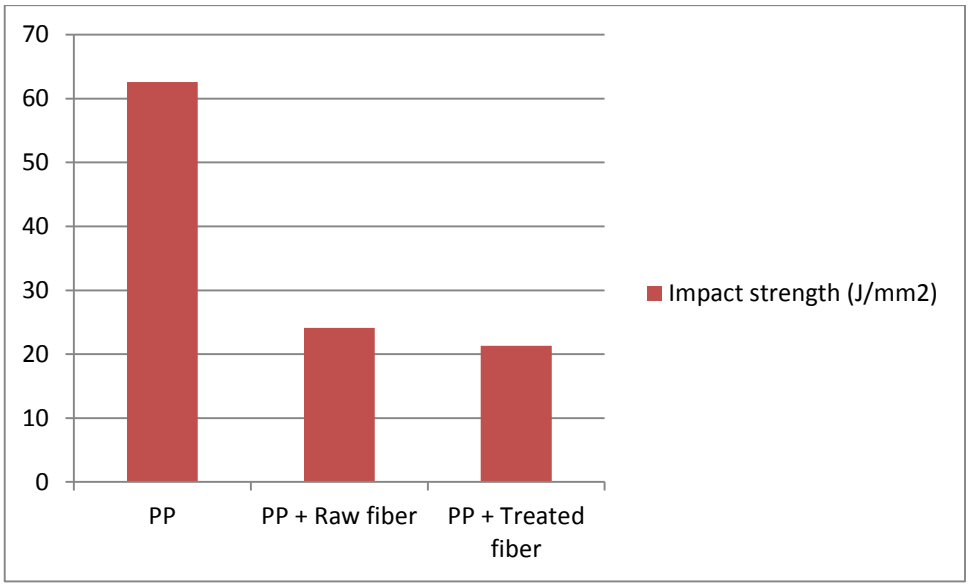

Fig. 4.44. Comparison of impact strength of materials

Impact strength, also known as impact energy, represents the ability of a material to withstand fracture or the resistance of a material to mechanical shock. It is a measure of the energy required to fracture a material, or the amount of energy the material can absorb before it fractures. Materials with higher modulus or stiffness usually show lower impact resistance than more elastic materials [12]. Therefore impact strength of materials decrease as the modulus increase. This trend can be observed in the above composites of PP. PP has relatively higher resistance than its composites. The composite of raw fiber has a slightly higher impact resistance than that of the composite of treated fiber. This is due to the increased stiffness or modulus induced by the fiber reinforcements, and the further increase in stiffness due to fiber pretreatment.

4.3. Physical properties of composite

4.3.1. Density

The density values and percentage changes occurring as a result of reinforcement are given in table 4.9. The percentage change indicates the effect of the fiber on the density of the original polymer, and also the effect of pretreatment of the fiber on this property of the composite.

Table 4.9. Density of materials

Material	Density (g/cm^3)	Percentage change
Polypropylene	0.84	
PP + raw fiber	0.88	3.86% increase
PP + treated fiber	0.83	4.65% decrease from PP+raw fiber 0.97% decrease from normal PP

Density is a measure of the weight per unit volume of material. The density of material depends on the composition of that material, and for a composite, it depends on the densities of the different materials in the composite. Figure 4.45 is a bar chart of the density of the polymer and the composites.

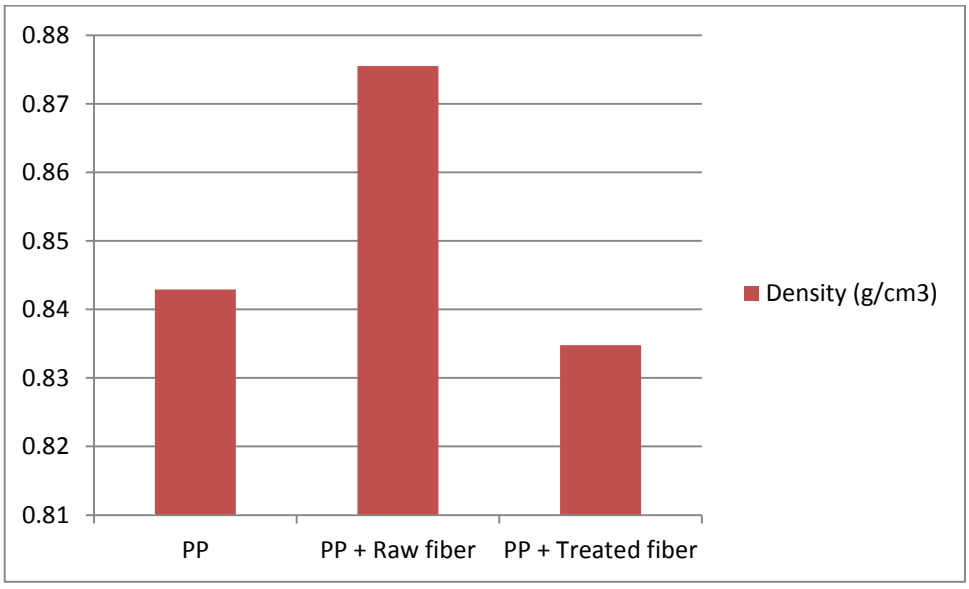

Fig. 4.45. Comparison of density of materials

The density of PP was increased by 3.864% after the addition of raw fibers. PP composite made with treated fibers however showed a reduction in density –a decrease of 0.967% from the density value of normal PP, and 4.651% decrease from the density of the composite produced with raw fibers. The increase in density obtained after reinforcement with raw fibers is due to the introduction of the fibers whose natural contents, such as cellulose and lignin, confers on it a high density. After treatments, however, some of these natural contents are removed resulting in lighter fibers. This change is invariably reflected in the

density of the composite made with the treated fibers. This relates to the reported reduction in weight of fibers due to chemical treatments [18].

4.3.2. Moisture resistance

Moisture resistance means the resistance to moisture or water uptake by the polymer or composite. Here it represented by the percentage water absorption, whereby the least water absorption imply the highest moisture resistance. The water absorption occurring as a result of reinforcements are given in table 4.10. The percentage change indicates the effect of the fiber on the moisture resistance of the original polymer, and also the effect of pretreatment of the fiber on this property of the composite.

Table 4.10. Moisture resistance of materials

Material	Water absorption (%)	Percentage change
Polypropylene	0.34	
PP + raw fiber	1.65	388.9% increase
PP + treated fiber	1.61	377.3% increase 2.264% decrease from PP+raw fiber

Water absorption varies according to the type of material. Figure 4.46 is a bar chart of the moisture resistance of the respective materials.

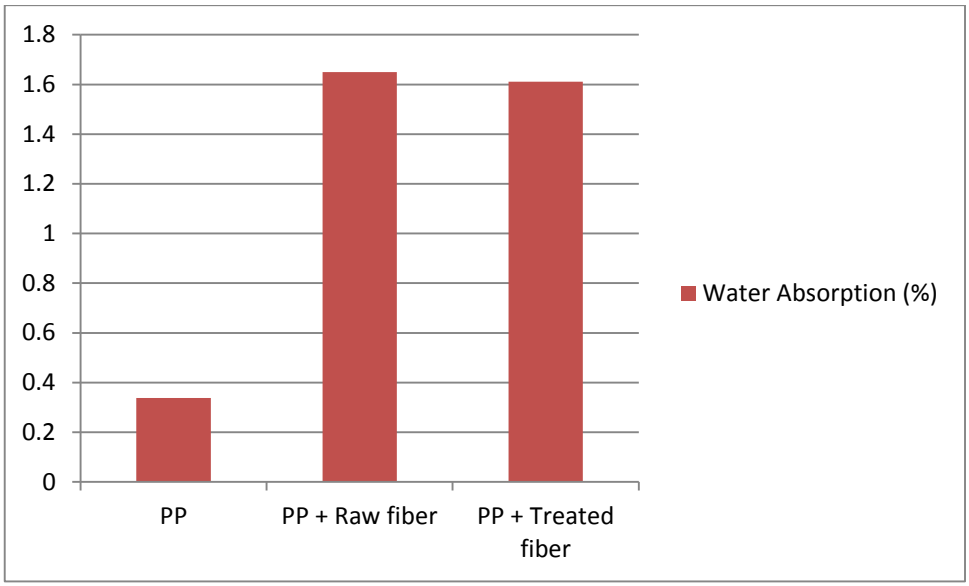

Fig. 4.46. Comparison of moisture resistance of materials

Reinforcement of PP with raw fibers resulted in increased water absorption of the composite. The moisture resistance of the composite was, however, slightly improved by the pretreatment of fibers as evidenced by the slight reduction (2.264%) in water absorption.

Natural fibers are generally hydrophilic and this explains the sharp rise in the value of percentage water absorption of the polymer reinforced with the fibers. Chemical treatment, on the other hand, decreases water uptake by modifying the chemical and physical structure of the fibers, thereby making the composite produced with treated fibers slightly more hydrophobic [25, 36, 37].

4.4. Scanning electron microscopy of composite

The images taken from the scanning electron microscope gave some details of the composite cross-section and fiber-matrix interaction. Figure 4.47 shows the fiber-polymer interphase inside the composite. This shows poor adhesion between the fiber and the polymer matrix as depicted by the wide and loose gap in the interphase. In some case, there was outright fiber pull-out during the tensile loading as shown in figure 4.48.

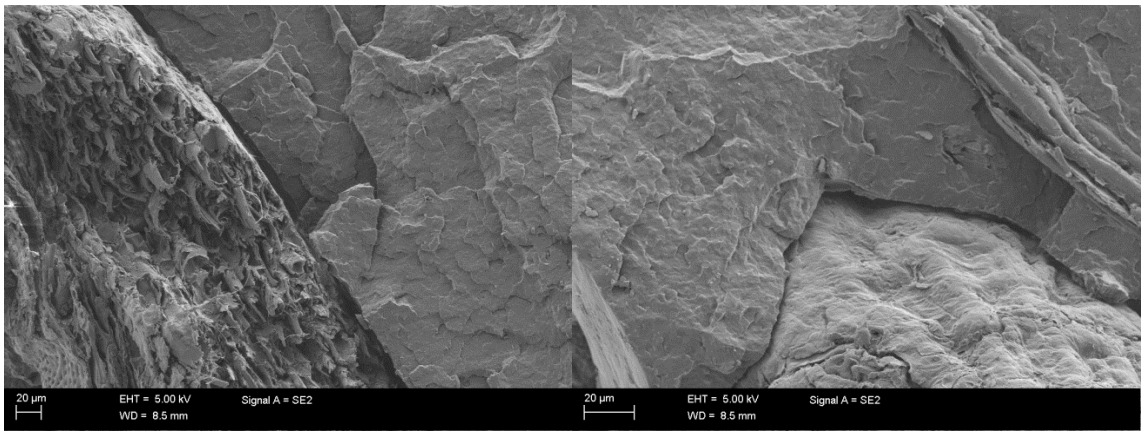

Fig. 4.47. SEM image of PP reinforced with raw fibers

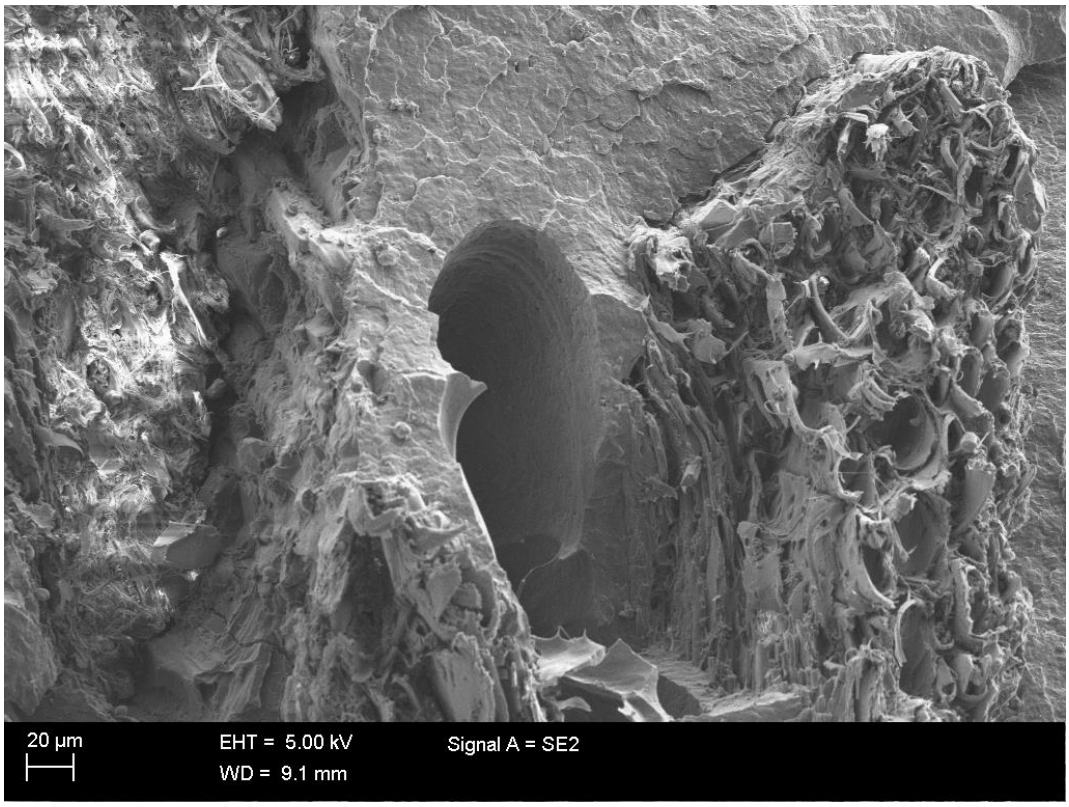

Fig. 4.48. Fiber pull-out hole in PP reinforced with raw fibers

On the other hand, treated fibers bonded better with polymer matrix as shown in figure 4.49. The gap between fiber and matrix is very minimal or non-existent. There were also no fiber pull-outs. This is in concordance with the general belief that fiber surface treatment enhances adhesion between natural fibers and polymer in a composite. The adhesion is promoted by the chemical reaction between the polymer and active sites of the treated fiber, as well as the enhanced mechanical interlocking of the polymer with the delignified fibers.

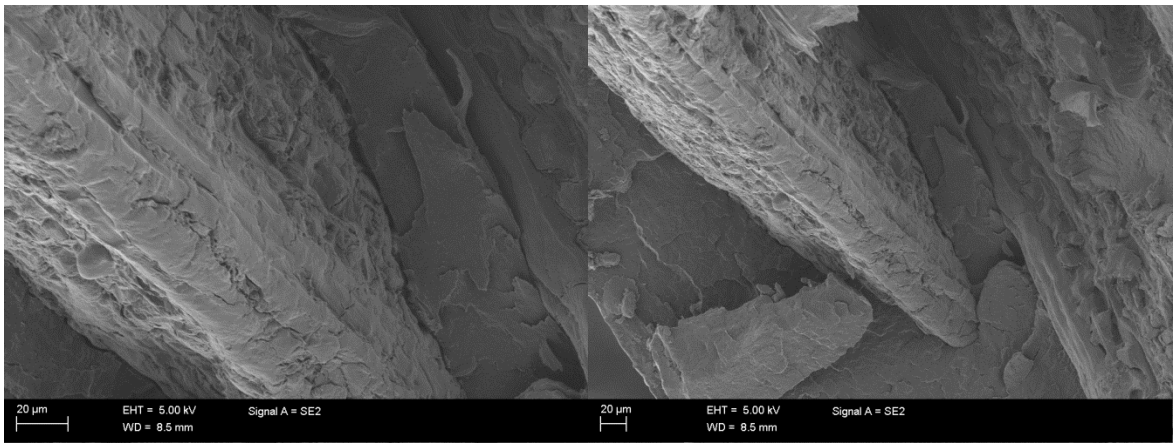

*Fig. 4.49. SEM image of PP reinforced with treated fiber*s

4.5. Reliability of the research

All tests used in this research were performed according to mentioned standards while referring to previous similar researches for more guidelines. The materials used were sourced from reputed a chemical company and hence the specifications were considered accurate. The experiments were performed in well-equipped laboratories under standard conditions. The data were recorded and properly analyzed in popular statistical software –excel and minitab. Hence the results can be considered reliable.

Possible sources of errors exist and are specific to individual tests. The major source of error could be found in fiber tensile tests where there were irregularities in the structure of the fiber and these affected the maximum and yield strength. The standard deviation of the tensile strength was 150 MPa for treated (NaOH 5%-1hour) fibers and 397.5 MPa for the raw fibers. In fiber and composite water absorption tests, evaporation rapidly occurred on exposure during weighing of the materials, slightly affecting the accuracy of wet weights.

For composite mechanical tests, presence of short fibers in the polymer introduced non-uniformity in the polymer body and in some cases, the composite failed at points other than the expected point of failure. The use of parallel samples largely helped to address these errors but some deviations are still possible. The composite tensile strength and modulus values are not up to expectation. Possible reasons are that the fibers degenerated due to the heat and shear encountered during composite production, or that the fibers, which mostly exist as semi-rigid fiber bundles, created crack initiation site at either end of the fiber where adhesion is minimal. In this case, the cracks could have led to premature failure.

5. CONCLUSION

Momordica Angustisepala has been studied to determine the possibility of using it as reinforcement in polymer matrices in composite production. Many other bast fibers have been used for similar role as had been captured in the literature review. The issue of fiber-matrix adhesion was first handled by treating the fiber with various chemical and studying the effect of these treatments on the properties and structure of the fiber. Then, the fibers were used in producing composites and the properties of the composites were equally studied to determine the efficacy of the fiber as a reinforcing material.

The fibers were treated with the following chemicals: sodium hydroxide, potassium hydroxide, silane, dicumyl peroxide, maleic anhydride and sodium hypochlorite. The fibers were tested for moisture content, water absorption, density and tensile properties. Sodium hydroxide treatment was determined to have the overall best effect on the fibers, and it was selected for the treatment of fibers that would be used for composite production. All fiber treatment methods showed decreased water absorption and density with increasing concentration and time of treatment, but the tensile properties were used to make the selection. One chemical treatment was selected such that it would give an optimal tensile strength and modulus while maintaining minimal water absorption and density of fiber. Sodium hydroxide solution of 5% concentration and 1 hour treatment time satisfied these criteria. Sodium hydroxide treatment showed a tensile strength of over 600MPa and a Young's modulus of over 3000MPa. Using scanning electron microscopy, images were obtained of fibers treated with the chemicals. Different degrees of changes were observed in the morphology of the treated fibers. Fibers treated with NaOH, KOH and silane showed groove-like features on the surface, DCP-treated fiber was slightly disfigured while MA-treated and NaOCl-treated fibers showed very minimal changes.

Polypropylene was used as the matrix in two types of composite –one with raw fiber and one with treated fibers. The fibers were cut short and 10% weight of the fibers was used in each composite. The production was done using compression moulding, preceded by high shear mixing. Samples of the polymer and the composites were studied for these properties: tensile strength, tensile modulus, strain, flexural strength, flexural modulus, impact strength, density and moisture resistance. Table 5.1 shows all the results determined from the experiments.

Table 5.1. Summary of the properties of polypropylene and its composites

Property	Polypropylene	Raw-fiber composite	Treated-fiber composite
Tensile strength (MPa)	28.49	18.79	21.57
Young's modulus (MPa)	1093.12	1080.63	1115.37
Elastic strain (mm/mm)	0.08	0.05	0.05
Flexural strength (MPa)	38.89	37.88	36.19
Flexural modulus (MPa)	1820	1800.15	1480.82
Impact strength J/mm^2	62.59	24.14	21.34
Density (g/cm^3)	0.843	0.88	0.84
Water absorption (%)	0.34	1.65	1.61

Improvement was only achieved in Young's modulus of the polymer. The Young's modulus of polypropylene was slightly improved when combined with treated fibers. The tensile strength values of the composites were lower than that of the polymer, but treated-fiber composite was stronger than the raw-fiber composite. The elastic strain was reduced by the presence of the fibers. The flexural strength, flexural modulus and impact strength were equally not increased by the addition of the fibers. The density of raw-fiber composite was slightly higher than the polymer's because the fibers are heavier than the polymer. The use of treated fibers helped to lower the density of the composite to a value below that of the polymer. Water absorption was drastically increased by the addition of fibers, but was less in the treated-fiber composite.

Composites of the fiber can be used as replacements for PP in applications where good elastic modulus or stiffness needs to be retained, but not where other mechanical properties are equally considered important. They can be used as replacements for wood in structural applications, especially where wood encounters problem arising from moisture absorption.

Applications that should particularly be avoided for this type of composite are applications that bear frequent impact load or force. These fibers considerably lower the impact resistance of the original polymer, even though this implies that the stiffness is equally increased as impact strength and stiffness have an indirect relationship. Hence, stiffness or Young's modulus remains the most prominent property of this new composite. The cost of the fiber is also an important factor since its low cost would help to bring down the cost of normal polymers used for suitable applications.

The low values that were obtained for most mechanical properties must have resulted from a number of factors. Paramount among these is the weight or volume percent of the fibers used for the reinforcement. 10%-wt. could be too low for any significant improvement. Future researches could study the effects when other volume or weight fractions are used for producing the composite. Another factor is the type of polymer used. Polypropylene is a thermoplastic; it would be interesting to know the effect of these fibers on the properties of thermosets such as polyester or epoxy. The processing method and conditions may have contributed to the resulting properties of the composites. High temperature process was used in melting the polymer and mixing it with the fiber; the effect of this on the fiber is unknown. It is possible that these natural fibers were denatured and made weaker by the process. Lastly, the length of the fibers could also have affected the composite properties. It was noticed that the short fibers used were also cut shorter by the high-shear mixer during the processing stage. Future studies could use other processing methods, or use longer fibers, to determine the effect these would have on the mechanical properties of the composite.

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APPENDIX A: NAOH-TREATED FIBERS –DATA AND CENTRAL COMPOSITE DESIGN

StdOrder	RunOrder	PtType	Blocks	Concentra	Time (hrs)	Strength MPa	Modulus MPa	Density g/cm3	Absortion %
1	1	1	1	0	0	183.2071682	1942.880548	1.114382388	92.03747073
2	2	1	1	10	0	133.8498601	940.0031651	1.104432304	55.64202335
3	3	1	1	0	6	83.16182463	444.1959873	0.965526129	81.74962293
4	4	1	1	10	6	32.59670435	186.6197241	0.416706951	85.80542265
5	5	-1	1	-2.07107	3	182.2051834	981.8534915	1.323432184	79.15690867
6	6	-1	1	12.07107	3	81.92869505	303.5157522	0.531998596	90.48543689
7	7	-1	1	5	-1.24264	210.5475831	828.1099371	1.770058643	91.05613701
8	8	-1	1	5	7.242641	62.40146626	217.6441086	0.56619504	72.11428571
9	9	0	1	5	3	255.1129566	1084.588024	2.366629093	66.37931034
10	10	0	1	5	3	127.8088867	599.8719784	1.104484198	61.0989011
11	11	0	1	5	3	135.7413931	527.315178	1.019522005	49.49402024
12	12	0	1	5	3	159.7301309	901.6762946	1.365946932	65.91928251
13	13	0	1	5	3	52.45989122	320.9665179	0.565350584	55.30612245

Central Composite Design

```
Factors:       2     Replicates:     1
Base runs:    13     Total runs:    13
Base blocks:   1     Total blocks:   1

Two-level factorial: Full factorial

Cube points:             4
Center points in cube:   5
Axial points:            4
Center points in axial:  0

α: 1.41421
```

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Welcome to Minitab, press F1 for help.
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Response Surface Regression: Strength MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	30332.6	6066.5	1.86	0.221
Linear	2	28399.9	14200.0	4.35	0.059
Concentration (%)	1	7304.5	7304.5	2.24	0.178
Time (hrs)	1	21095.5	21095.5	6.46	0.039
Square	2	1932.3	966.1	0.30	0.753
Concentration (%)*Concentration (%)	1	1280.7	1280.7	0.39	0.551
Time (hrs)*Time (hrs)	1	898.5	898.5	0.28	0.616
2-Way Interaction	1	0.4	0.4	0.00	0.992
Concentration (%)*Time (hrs)	1	0.4	0.4	0.00	0.992
Error	7	22866.7	3266.7		
Lack-of-Fit	3	1586.7	528.9	0.10	0.956
Pure Error	4	21279.9	5320.0		
Total	12	53199.2			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
57.1547	57.02%	26.31%	16.29%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		146.2	25.6	5.72	0.001	
Concentration (%)	-60.4	-30.2	20.2	-1.50	0.178	1.00

Time (hrs)	-102.7	-51.4	20.2	-2.54	0.039	1.00
Concentration (%)*Concentration (%)	-27.1	-13.6	21.7	-0.63	0.551	1.02
Time (hrs)*Time (hrs)	-22.7	-11.4	21.7	-0.52	0.616	1.02
Concentration (%)*Time (hrs)	-0.6	-0.3	28.6	-0.01	0.992	1.00

Regression Equation in Uncoded Units

Strength MPa = 202.5 - 0.6 Concentration (%) - 9.4 Time (hrs) - 0.543 Concentration (%) *Concentration (%) - 1.26 Time (hrs)*Time (hrs) - 0.02 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Strength MPa	Fit	Resid	Std Resid	
9	255.1	146.2	108.9	2.13	R

R Large residual

Response Surface Regression: Modulus MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1988237	397647	3.49	0.067
Linear	2	1829134	914567	8.02	0.015
Concentration (%)	1	615921	615921	5.40	0.053
Time (hrs)	1	1213212	1213212	10.64	0.014
Square	2	20235	10118	0.09	0.916
Concentration (%)*Concentration (%)	1	18674	18674	0.16	0.698
Time (hrs)*Time (hrs)	1	456	456	0.00	0.951
2-Way Interaction	1	138868	138868	1.22	0.306
Concentration (%)*Time (hrs)	1	138868	138868	1.22	0.306
Error	7	798454	114065		
Lack-of-Fit	3	427221	142407	1.53	0.336
Pure Error	4	371233	92808		
Total	12	2786691			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
337.735	71.35%	50.88%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		687	151	4.55	0.003	
Concentration (%)	-555	-277	119	-2.32	0.053	1.00
Time (hrs)	-779	-389	119	-3.26	0.014	1.00
Concentration (%)*Concentration (%)	104	52	128	0.40	0.698	1.02
Time (hrs)*Time (hrs)	-16	-8	128	-0.06	0.951	1.02
Concentration (%)*Time (hrs)	373	186	169	1.10	0.306	1.00

Regression Equation in Uncoded Units

Modulus MPa = 1584 - 113.5 Concentration (%) - 187 Time (hrs) + 2.07 Concentration (%) *Concentration (%) - 0.9 Time (hrs)*Time (hrs) + 12.4 Concentration (%) *Time (hrs)

Response Surface Regression: Density g/cm3 versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1.58450	0.31690	1.12	0.428
Linear	2	1.15785	0.57893	2.05	0.199
Concentration (%)	1	0.35197	0.35197	1.25	0.301
Time (hrs)	1	0.80588	0.80588	2.86	0.135
Square	2	0.35405	0.17703	0.63	0.561
Concentration (%)*Concentration (%)	1	0.32231	0.32231	1.14	0.321

Time (hrs)*Time (hrs)	1	0.06284	0.06284	0.22	0.651
2-Way Interaction	1	0.07259	0.07259	0.26	0.627
Concentration (%)*Time (hrs)	1	0.07259	0.07259	0.26	0.627
Error	7	1.97404	0.28201		
Lack-of-Fit	3	0.17661	0.05887	0.13	0.937
Pure Error	4	1.79743	0.44936		
Total	12	3.55854			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.531042	44.53%	4.90%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		1.284	0.237	5.41	0.001	
Concentration (%)	-0.420	-0.210	0.188	-1.12	0.301	1.00
Time (hrs)	-0.635	-0.317	0.188	-1.69	0.135	1.00
Concentration (%)*Concentration (%)	-0.431	-0.215	0.201	-1.07	0.321	1.02
Time (hrs)*Time (hrs)	-0.190	-0.095	0.201	-0.47	0.651	1.02
Concentration (%)*Time (hrs)	-0.269	-0.135	0.266	-0.51	0.627	1.00

Regression Equation in Uncoded Units

Density g/cm3 = 1.367 + 0.071 Concentration (%) + 0.002 Time (hrs)
- 0.00861 Concentration (%)*Concentration (%) -
0.0106 Time (hrs)*Time (hrs)
- 0.0090 Concentration (%)*Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Density g/cm3	Fit	Resid	Std Resid	
9	2.367	1.284	1.082	2.28	R

R Large residual

Response Surface Regression: Absortion % versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1863.49	372.698	3.21	0.080
Linear	2	39.26	19.630	0.17	0.848
Concentration (%)	1	33.29	33.287	0.29	0.609
Time (hrs)	1	5.97	5.972	0.05	0.827
Square	2	1415.16	707.578	6.10	0.029
Concentration (%)*Concentration (%)	1	918.75	918.750	7.92	0.026
Time (hrs)*Time (hrs)	1	678.26	678.260	5.85	0.046
2-Way Interaction	1	409.08	409.076	3.53	0.102
Concentration (%)*Time (hrs)	1	409.08	409.076	3.53	0.102
Error	7	811.85	115.979		
Lack-of-Fit	3	603.15	201.051	3.85	0.113
Pure Error	4	208.70	52.175		
Total	12	2675.34			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
10.7693	69.65%	47.98%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		59.64	4.82	12.38	0.000	
Concentration (%)	-4.08	-2.04	3.81	-0.54	0.609	1.00
Time (hrs)	-1.73	-0.86	3.81	-0.23	0.827	1.00
Concentration (%)*Concentration (%)	22.98	11.49	4.08	2.81	0.026	1.02
Time (hrs)*Time (hrs)	19.75	9.87	4.08	2.42	0.046	1.02

Concentration (%)*Time (hrs)	20.23	10.11	5.38	1.88	0.102	1.00

Regression Equation in Uncoded Units

Absorption % = 94.02 - 7.03 Concentration (%) - 10.24 Time (hrs) + 0.460 Concentration (%) *Concentration (%) + 1.097 Time (hrs)*Time (hrs) + 0.674 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Absorption %	Fit	Resid	Std Resid	
2	55.64	69.72	-14.08	-2.13	R

R Large residual

APPENDIX B: KOH-TREATED FIBER –DATA AND CENTRAL COMPOSITE DESIGN

StdOrder	RunOrder	PtType	Blocks	Concentra	Time (hrs)	Strength MPa	Modulus MPa	Density g/cm3	Absortion %
1	1	1	1	0	0	183.2071682	1942.880548	1.114382388	92.03747073
2	2	1	1	10	0	28.716083	163.5437939	0.303673878	71.42857143
3	3	1	1	0	6	128.5684064	619.5635766	1.061580761	72.78225806
4	4	1	1	10	6	58.03786108	371.5452512	0.517966591	73.01038062
5	5	-1	1	-2.07107	3	71.02465378	459.2255826	1.142124156	81.30165289
6	6	-1	1	12.07107	3	122.7977393	538.0059211	1.080650458	76.55172414
7	7	-1	1	5	-1.24264	164.9396866	779.9235869	1.069417942	83.14606742
8	8	-1	1	5	7.242641	201.5996641	894.6011788	1.43723298	82.66384778
9	9	0	1	5	3	129.825349	511.9646865	0.863191281	87.61061947
10	10	0	1	5	3	124.8172951	663.2750626	1.090349652	65.65656566
11	11	0	1	5	3	89.49237934	594.1106014	1.103076236	55.79514825
12	12	0	1	5	3	128.2739299	822.2616979	1.282354189	64.07263294
13	13	0	1	5	3	65.20197528	596.7812178	0.691887731	34.4765343

Central Composite Design

```
Factors:       2     Replicates:     1
Base runs:    13     Total runs:    13
Base blocks:   1     Total blocks:   1

Two-level factorial: Full factorial

Cube points:             4
Center points in cube:   5
Axial points:            4
Center points in axial:  0

α: 1.41421
```

Response Surface Regression: Strength MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	12657.1	2531.42	0.96	0.501
Linear	2	2968.5	1484.25	0.56	0.594
Concentration (%)	1	2880.5	2880.54	1.09	0.331
Time (hrs)	1	88.0	87.97	0.03	0.860
Square	2	7926.3	3963.13	1.50	0.287
Concentration (%)*Concentration (%)	1	1654.1	1654.10	0.63	0.455
Time (hrs)*Time (hrs)	1	5360.5	5360.52	2.03	0.197
2-Way Interaction	1	1762.3	1762.34	0.67	0.441
Concentration (%)*Time (hrs)	1	1762.3	1762.34	0.67	0.441
Error	7	18479.6	2639.94		
Lack-of-Fit	3	15136.3	5045.44	6.04	0.058
Pure Error	4	3343.3	835.82		
Total	12	31136.7			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
51.3804	40.65%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		107.5	23.0	4.68	0.002	
Concentration (%)	-38.0	-19.0	18.2	-1.04	0.331	1.00
Time (hrs)	6.6	3.3	18.2	0.18	0.860	1.00
Concentration (%)*Concentration (%)	-30.8	-15.4	19.5	-0.79	0.455	1.02
Time (hrs)*Time (hrs)	55.5	27.8	19.5	1.42	0.197	1.02
Concentration (%)*Time (hrs)	42.0	21.0	25.7	0.82	0.441	1.00

Regression Equation in Uncoded Units

Strength MPa = 156.5 - 1.8 Concentration (%) - 24.4 Time (hrs) - 0.617 Concentration (%) *Concentration (%) + 3.08 Time (hrs)*Time (hrs) + 1.40 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Strength MPa	Fit	Resid	Std Resid	
4	58.0	125.2	-67.2	-2.13	R
6	122.8	49.8	73.0	2.32	R

R Large residual

Response Surface Regression: Modulus MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1294773	258955	2.13	0.177
Linear	2	572413	286207	2.35	0.166
Concentration (%)	1	458855	458855	3.77	0.093
Time (hrs)	1	113559	113559	0.93	0.366
Square	2	136126	68063	0.56	0.595
Concentration (%)*Concentration (%)	1	12815	12815	0.11	0.755
Time (hrs)*Time (hrs)	1	111149	111149	0.91	0.371
2-Way Interaction	1	586234	586234	4.81	0.064
Concentration (%)*Time (hrs)	1	586234	586234	4.81	0.064
Error	7	852551	121793		
Lack-of-Fit	3	798450	266150	19.68	0.007
Pure Error	4	54101	13525		
Total	12	2147324			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
348.989	60.30%	31.94%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		638	156	4.09	0.005	
Concentration (%)	-479	-239	123	-1.94	0.093	1.00
Time (hrs)	-238	-119	123	-0.97	0.366	1.00
Concentration (%)*Concentration (%)	-86	-43	132	-0.32	0.755	1.02
Time (hrs)*Time (hrs)	253	126	132	0.96	0.371	1.02
Concentration (%)*Time (hrs)	766	383	174	2.19	0.064	1.00

Regression Equation in Uncoded Units

Modulus MPa = 1463 - 107.3 Concentration (%) - 252 Time (hrs) - 1.72 Concentration (%) *Concentration (%) + 14.0 Time (hrs)*Time (hrs) + 25.5 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Modulus MPa	Fit	Resid	Std Resid	
1	1943	1463	480	2.25	R
5	459	891	-431	-2.02	R

R Large residual

Response Surface Regression: Density g/cm3 versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	0.36071	0.072143	0.63	0.685
Linear	2	0.31774	0.158868	1.38	0.312

Concentration (%)	1	0.25965	0.259654	2.26	0.176
Time (hrs)	1	0.05808	0.058083	0.51	0.500
Square	2	0.02514	0.012571	0.11	0.898
Concentration (%)*Concentration (%)	1	0.02153	0.021529	0.19	0.678
Time (hrs)*Time (hrs)	1	0.00164	0.001637	0.01	0.908
2-Way Interaction	1	0.01783	0.017835	0.16	0.705
Concentration (%)*Time (hrs)	1	0.01783	0.017835	0.16	0.705
Error	7	0.80374	0.114819		
Lack-of-Fit	3	0.59177	0.197255	3.72	0.118
Pure Error	4	0.21197	0.052993		
Total	12	1.16445			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.338850	30.98%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		1.006	0.152	6.64	0.000	
Concentration (%)	-0.360	-0.180	0.120	-1.50	0.176	1.00
Time (hrs)	0.170	0.085	0.120	0.71	0.500	1.00
Concentration (%)*Concentration (%)	-0.111	-0.056	0.128	-0.43	0.678	1.02
Time (hrs)*Time (hrs)	0.031	0.015	0.128	0.12	0.908	1.02
Concentration (%)*Time (hrs)	0.134	0.067	0.169	0.39	0.705	1.00

Regression Equation in Uncoded Units

Density g/cm3 = 1.128 - 0.0271 Concentration (%) - 0.004 Time (hrs)
- 0.00223 Concentration (%)*Concentration (%)
+ 0.0017 Time (hrs)*Time (hrs)
+ 0.0045 Concentration (%)*Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Density g/cm3	Fit	Resid	Std Resid	
4	0.518	0.938	-0.420	-2.02	R
6	1.081	0.640	0.441	2.12	R

R Large residual

Response Surface Regression: Absortion % versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1210.62	242.12	1.09	0.441
Linear	2	133.90	66.95	0.30	0.749
Concentration (%)	1	91.79	91.79	0.41	0.541
Time (hrs)	1	42.11	42.11	0.19	0.676
Square	2	968.17	484.09	2.18	0.184
Concentration (%)*Concentration (%)	1	423.44	423.44	1.91	0.210
Time (hrs)*Time (hrs)	1	666.88	666.88	3.00	0.127
2-Way Interaction	1	108.55	108.55	0.49	0.507
Concentration (%)*Time (hrs)	1	108.55	108.55	0.49	0.507
Error	7	1553.83	221.98		
Lack-of-Fit	3	85.36	28.45	0.08	0.969
Pure Error	4	1468.47	367.12		
Total	12	2764.45			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
14.8989	43.79%	3.64%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF

Constant		61.52	6.66	9.23	0.000	
Concentration (%)	-6.77	-3.39	5.27	-0.64	0.541	1.00
Time (hrs)	-4.59	-2.29	5.27	-0.44	0.676	1.00
Concentration (%)*Concentration (%)	15.60	7.80	5.65	1.38	0.210	1.02
Time (hrs)*Time (hrs)	19.58	9.79	5.65	1.73	0.127	1.02
Concentration (%)*Time (hrs)	10.42	5.21	7.45	0.70	0.507	1.00

Regression Equation in Uncoded Units

Absortion % = 90.0 - 4.84 Concentration (%) - 9.03 Time (hrs) + 0.312 Concentration (%) *Concentration (%) + 1.088 Time (hrs)*Time (hrs) + 0.347 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Absortion %	Fit	Resid	Std Resid	
13	34.48	61.52	-27.05	-2.03	R

R Large residual

APPENDIX C: SILANE-TREATED FIBER –DATA AND CENTRAL COMPOSITE DESIGN

StdOrder	RunOrder	PtType	Blocks	Concentra	Time (hrs)	Strength MPa	Modulus MPa	Density g/cm3	Absortion %
1	1	1	1	0	0	183.2071682	1942.880548	1.114382388	92.03747073
2	2	1	1	5	0	64.22050619	313.4905255	0.402152271	55.40897098
3	3	1	1	0	3	132.7484418	874.8097017	0.888366624	59.63541667
4	4	1	1	5	3	80.11806873	576.3033335	0.635874038	51.87165775
5	5	-1	1	-1.03553	1.5	35.85023115	167.0009199	0.310951641	71.22641509
6	6	-1	1	6.035534	1.5	76.69542833	342.195509	0.642055316	60.73825503
7	7	-1	1	2.5	-0.62132	65.61087683	348.9688624	0.553479541	63.40206186
8	8	-1	1	2.5	3.62132	53.57405743	415.1026095	0.545095081	73.79454927
9	9	0	1	2.5	1.5	59.18058257	370.1564554	0.53420926	63.17204301
10	10	0	1	2.5	1.5	169.5388729	1134.674801	1.234696992	58.41346154
11	11	0	1	2.5	1.5	137.9890173	770.8381949	1.187109063	51.64179104
12	12	0	1	2.5	1.5	59.63228306	494.4304096	0.338618779	59.84042553
13	13	0	1	2.5	1.5	85.79742686	588.9591539	0.532294443	46.7268623

Central Composite Design

```
Factors:       2     Replicates:     1
Base runs:    13     Total runs:    13
Base blocks:   1     Total blocks:   1

Two-level factorial: Full factorial

Cube points:             4
Center points in cube:   5
Axial points:            4
Center points in axial:  0

α: 1.41421
```

Response Surface Regression: Strength MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	3845.0	769.0	0.24	0.935
Linear	2	1952.9	976.5	0.30	0.751
Concentration (%)	1	1620.3	1620.3	0.50	0.504
Time (hrs)	1	332.6	332.6	0.10	0.759
Square	2	791.3	395.6	0.12	0.888
Concentration (%)*Concentration (%)	1	537.8	537.8	0.16	0.697
Time (hrs)*Time (hrs)	1	353.9	353.9	0.11	0.752
2-Way Interaction	1	1100.8	1100.8	0.34	0.580
Concentration (%)*Time (hrs)	1	1100.8	1100.8	0.34	0.580
Error	7	22892.4	3270.3		
Lack-of-Fit	3	13145.6	4381.9	1.80	0.287
Pure Error	4	9746.8	2436.7		
Total	12	26737.4			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
57.1869	14.38%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		102.4	25.6	4.01	0.005	
Concentration (%)	-28.5	-14.2	20.2	-0.70	0.504	1.00
Time (hrs)	-12.9	-6.4	20.2	-0.32	0.759	1.00
Concentration (%)*Concentration (%)	-17.6	-8.8	21.7	-0.41	0.697	1.02

Time (hrs)*Time (hrs)	-14.3	-7.1	21.7	-0.33	0.752	1.02
Concentration (%)*Time (hrs)	33.2	16.6	28.6	0.58	0.580	1.00

Regression Equation in Uncoded Units

Strength MPa = 123.8 - 5.3 Concentration (%) - 5.8 Time (hrs) - 1.41 Concentration (%) *Concentration (%) - 3.17 Time (hrs)*Time (hrs) + 4.42 Concentration (%) *Time (hrs)

Response Surface Regression: Modulus MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	882683	176537	0.69	0.646
Linear	2	416176	208088	0.82	0.481
Concentration (%)	1	352856	352856	1.38	0.278
Time (hrs)	1	63320	63320	0.25	0.634
Square	2	23694	11847	0.05	0.955
Concentration (%)*Concentration (%)	1	22182	22182	0.09	0.777
Time (hrs)*Time (hrs)	1	366	366	0.00	0.971
2-Way Interaction	1	442813	442813	1.73	0.229
Concentration (%)*Time (hrs)	1	442813	442813	1.73	0.229
Error	7	1786712	255245		
Lack-of-Fit	3	1433339	477780	5.41	0.068
Pure Error	4	353373	88343		
Total	12	2669395			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
505.217	33.07%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		672	226	2.97	0.021	
Concentration (%)	-420	-210	179	-1.18	0.278	1.00
Time (hrs)	-178	-89	179	-0.50	0.634	1.00
Concentration (%)*Concentration (%)	-113	-56	192	-0.29	0.777	1.02
Time (hrs)*Time (hrs)	15	7	192	0.04	0.971	1.02
Concentration (%)*Time (hrs)	665	333	253	1.32	0.229	1.00

Regression Equation in Uncoded Units

Modulus MPa = 1254 - 172 Concentration (%) - 291 Time (hrs) - 9.0 Concentration (%) *Concentration (%) + 3.2 Time (hrs)*Time (hrs) + 88.7 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Modulus MPa	Fit	Resid	Std Resid	
1	1943	1254	689	2.23	R
5	167	856	-689	-2.23	R

R Large residual

Response Surface Regression: Density g/cm3 versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	0.14003	0.028005	0.18	0.960
Linear	2	0.03081	0.015406	0.10	0.905
Concentration (%)	1	0.03081	0.030810	0.20	0.667
Time (hrs)	1	0.00000	0.000002	0.00	0.997
Square	2	0.05637	0.028187	0.18	0.835
Concentration (%)*Concentration (%)	1	0.04748	0.047482	0.31	0.594
Time (hrs)*Time (hrs)	1	0.01486	0.014864	0.10	0.764

2-Way Interaction	1	0.05284	0.052840	0.35	0.575
Concentration (%)*Time (hrs)	1	0.05284	0.052840	0.35	0.575
Error	7	1.06705	0.152435		
Lack-of-Fit	3	0.37904	0.126346	0.73	0.584
Pure Error	4	0.68801	0.172002		
Total	12	1.20707			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.390429	11.60%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		0.765	0.175	4.38	0.003	
Concentration (%)	-0.124	-0.062	0.138	-0.45	0.667	1.00
Time (hrs)	-0.001	-0.001	0.138	-0.00	0.997	1.00
Concentration (%)*Concentration (%)	-0.165	-0.083	0.148	-0.56	0.594	1.02
Time (hrs)*Time (hrs)	-0.092	-0.046	0.148	-0.31	0.764	1.02
Concentration (%)*Time (hrs)	0.230	0.115	0.195	0.59	0.575	1.00

Regression Equation in Uncoded Units

Density g/cm3 = 0.814 - 0.005 Concentration (%) - 0.015 Time (hrs) - 0.0132 Concentration (%) *Concentration (%) - 0.0205 Time (hrs)*Time (hrs) + 0.0306 Concentration (%) *Time (hrs)

Response Surface Regression: Absortion % versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1021.10	204.22	2.31	0.153
Linear	2	494.85	247.43	2.79	0.128
Concentration (%)	1	438.45	438.45	4.95	0.061
Time (hrs)	1	56.40	56.40	0.64	0.451
Square	2	317.96	158.98	1.80	0.235
Concentration (%)*Concentration (%)	1	133.07	133.07	1.50	0.260
Time (hrs)*Time (hrs)	1	224.57	224.57	2.54	0.155
2-Way Interaction	1	208.29	208.29	2.35	0.169
Concentration (%)*Time (hrs)	1	208.29	208.29	2.35	0.169
Error	7	619.74	88.53		
Lack-of-Fit	3	442.75	147.58	3.34	0.137
Pure Error	4	176.99	44.25		
Total	12	1640.84			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
9.40929	62.23%	35.25%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		55.96	4.21	13.30	0.000	
Concentration (%)	-14.81	-7.40	3.33	-2.23	0.061	1.00
Time (hrs)	-5.31	-2.66	3.33	-0.80	0.451	1.00
Concentration (%)*Concentration (%)	8.75	4.37	3.57	1.23	0.260	1.02
Time (hrs)*Time (hrs)	11.36	5.68	3.57	1.59	0.155	1.02
Concentration (%)*Time (hrs)	14.43	7.22	4.70	1.53	0.169	1.00

Regression Equation in Uncoded Units

Absortion % = 83.29 - 9.35 Concentration (%) - 14.16 Time (hrs) + 0.700 Concentration (%) *Concentration (%) + 2.53 Time (hrs)*Time (hrs) + 1.92 Concentration (%) *Time (hrs)

APPENDIX D: DCP-TREATED FIBER –DATA AND CENTRAL COMPOSITE DESIGN

StdOrder	RunOrder	PtType	Blocks	Concentra	Time (hrs)	Strength MPa	Modulus MPa	Density g/cm3	Absortion %
1	1	1	1	0	0	183.2071682	1942.880548	1.114382388	92.03747073
2	2	1	1	10	0	52.02263835	219.5559565	0.350798155	61.24031008
3	3	1	1	0	6	105.5049109	839.0924546	0.717744307	47.89915966
4	4	1	1	10	6	126.024436	812.390345	0.9783624	15.25129983
5	5	-1	1	-2.07107	3	46.48197733	228.5403518	0.426021632	54.52631579
6	6	-1	1	12.07107	3	88.63391895	398.4418389	0.570475692	10.30534351
7	7	-1	1	5	-1.24264	73.45193837	333.9912467	0.613547357	32.76955603
8	8	-1	1	5	7.242641	38.66851499	223.2588546	0.372239735	65.72164948
9	9	0	1	5	3	65.43419098	435.1432321	0.441064168	39.28571429
10	10	0	1	5	3	37.40742927	229.1161661	0.183985407	32.94117647
11	11	0	1	5	3	68.06168555	390.8515038	0.673285105	66.83250415
12	12	0	1	5	3	80.90886328	401.5823259	0.544288512	52.39085239
13	13	0	1	5	3	88.59635515	335.8056231	0.750847279	66.66666667

```
Central Composite Design

Factors:       2     Replicates:     1
Base runs:    13     Total runs:    13
Base blocks:   1     Total blocks:   1

Two-level factorial: Full factorial

Cube points:             4
Center points in cube:   5
Axial points:            4
Center points in axial:  0

α: 1.41421

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Welcome to Minitab, press F1 for help.
Retrieving project from file: 'C:\Users\hp\Documents\Minitab\sample.MPJ'

Response Surface Regression: Strength MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source                                  DF   Adj SS  Adj MS  F-Value  P-Value
Model                                    5   7939.9  1588.0     0.97    0.495
  Linear                                 2    675.5   337.7     0.21    0.818
    Concentration (%)                    1    325.8   325.8     0.20    0.669
    Time (hrs)                           1    349.7   349.7     0.21    0.658
  Square                                 2   1510.9   755.4     0.46    0.648
    Concentration (%)*Concentration (%)  1   1260.0  1260.0     0.77    0.409
    Time (hrs)*Time (hrs)                1    413.5   413.5     0.25    0.630
  2-Way Interaction                      1   5753.5  5753.5     3.52    0.103
    Concentration (%)*Time (hrs)         1   5753.5  5753.5     3.52    0.103
Error                                    7  11440.0  1634.3
  Lack-of-Fit                            3   9906.7  3302.2     8.61    0.032
  Pure Error                             4   1533.3   383.3
Total                                   12  19379.9


Model Summary

      S    R-sq  R-sq(adj)  R-sq(pred)
40.4264  40.97%      0.00%       0.00%


Coded Coefficients

Term                                  Effect  Coef  SE Coef  T-Value  P-Value   VIF
Constant                                      68.1     18.1     3.77    0.007
```

Concentration (%)	-12.8	-6.4	14.3	-0.45	0.669	1.00
Time (hrs)	-13.2	-6.6	14.3	-0.46	0.658	1.00
Concentration (%)*Concentration (%)	26.9	13.5	15.3	0.88	0.409	1.02
Time (hrs)*Time (hrs)	15.4	7.7	15.3	0.50	0.630	1.02
Concentration (%)*Time (hrs)	75.9	37.9	20.2	1.88	0.103	1.00

Regression Equation in Uncoded Units

Strength MPa = 140.2 - 14.24 Concentration (%) - 20.0 Time (hrs) + 0.538 Concentration (%) *Concentration (%) + 0.86 Time (hrs)*Time (hrs) + 2.53 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Strength MPa	Fit	Resid	Std Resid	
3	105.5	51.1	54.4	2.20	R
5	46.5	104.0	-57.5	-2.32	R

R Large residual

Response Surface Regression: Modulus MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1279621	255924	1.28	0.370
Linear	2	340621	170311	0.85	0.467
Concentration (%)	1	284918	284918	1.42	0.272
Time (hrs)	1	55703	55703	0.28	0.614
Square	2	219367	109684	0.55	0.601
Concentration (%)*Concentration (%)	1	139977	139977	0.70	0.431
Time (hrs)*Time (hrs)	1	107686	107686	0.54	0.487
2-Way Interaction	1	719632	719632	3.59	0.100
Concentration (%)*Time (hrs)	1	719632	719632	3.59	0.100
Error	7	1401298	200185		
Lack-of-Fit	3	1375266	458422	70.44	0.001
Pure Error	4	26032	6508		
Total	12	2680919			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
447.421	47.73%	10.40%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		358	200	1.79	0.116	
Concentration (%)	-377	-189	158	-1.19	0.272	1.00
Time (hrs)	-167	-83	158	-0.53	0.614	1.00
Concentration (%)*Concentration (%)	284	142	170	0.84	0.431	1.02
Time (hrs)*Time (hrs)	249	124	170	0.73	0.487	1.02
Concentration (%)*Time (hrs)	848	424	224	1.90	0.100	1.00

Regression Equation in Uncoded Units

Modulus MPa = 1321 - 179.3 Concentration (%) - 252 Time (hrs) + 5.67 Concentration (%) *Concentration (%) + 13.8 Time (hrs)*Time (hrs) + 28.3 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Modulus MPa	Fit	Resid	Std Resid	
1	1943	1321	622	2.27	R
5	229	909	-681	-2.48	R

R Large residual

Response Surface Regression: Density g/cm3 versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	0.322453	0.064491	0.95	0.504
Linear	2	0.012673	0.006336	0.09	0.912
Concentration (%)	1	0.011151	0.011151	0.16	0.697
Time (hrs)	1	0.001522	0.001522	0.02	0.885
Square	2	0.047532	0.023766	0.35	0.716
Concentration (%)*Concentration (%)	1	0.028019	0.028019	0.41	0.540
Time (hrs)*Time (hrs)	1	0.025705	0.025705	0.38	0.557
2-Way Interaction	1	0.262248	0.262248	3.88	0.090
Concentration (%)*Time (hrs)	1	0.262248	0.262248	3.88	0.090
Error	7	0.473712	0.067673		
Lack-of-Fit	3	0.277207	0.092402	1.88	0.274
Pure Error	4	0.196505	0.049126		
Total	12	0.796164			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.260141	40.50%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		0.519	0.116	4.46	0.003	
Concentration (%)	-0.0747	-0.0373	0.0920	-0.41	0.697	1.00
Time (hrs)	-0.0276	-0.0138	0.0920	-0.15	0.885	1.00
Concentration (%)*Concentration (%)	0.1269	0.0635	0.0986	0.64	0.540	1.02
Time (hrs)*Time (hrs)	0.1216	0.0608	0.0986	0.62	0.557	1.02
Concentration (%)*Time (hrs)	0.512	0.256	0.130	1.97	0.090	1.00

Regression Equation in Uncoded Units

Density g/cm3 = 0.950 - 0.0841 Concentration (%) - 0.1305 Time (hrs)
+ 0.00254 Concentration (%)*Concentration (%)
+ 0.0068 Time (hrs)*Time (hrs)
+ 0.01707 Concentration (%)*Time (hrs)

Response Surface Regression: Absortion % versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	2557.98	511.60	0.98	0.490
Linear	2	2220.78	1110.39	2.13	0.189
Concentration (%)	1	1983.96	1983.96	3.81	0.092
Time (hrs)	1	236.81	236.81	0.45	0.522
Square	2	336.35	168.17	0.32	0.734
Concentration (%)*Concentration (%)	1	274.76	274.76	0.53	0.491
Time (hrs)*Time (hrs)	1	31.57	31.57	0.06	0.813
2-Way Interaction	1	0.86	0.86	0.00	0.969
Concentration (%)*Time (hrs)	1	0.86	0.86	0.00	0.969
Error	7	3648.91	521.27		
Lack-of-Fit	3	2689.46	896.49	3.74	0.118
Pure Error	4	959.45	239.86		
Total	12	6206.90			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
22.8314	41.21%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		51.6	10.2	5.06	0.001	
Concentration (%)	-31.50	-15.75	8.07	-1.95	0.092	1.00

Time (hrs)	-10.88	-5.44	8.07	-0.67	0.522	1.00
Concentration (%)*Concentration (%)	-12.57	-6.28	8.66	-0.73	0.491	1.02
Time (hrs)*Time (hrs)	4.26	2.13	8.66	0.25	0.813	1.02
Concentration (%)*Time (hrs)	-0.9	-0.5	11.4	-0.04	0.969	1.00

Regression Equation in Uncoded Units

Absortion % = 68.2 - 0.54 Concentration (%) - 3.08 Time (hrs) - 0.251 Concentration (%) *Concentration (%) + 0.237 Time (hrs)*Time (hrs) - 0.031 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Absortion %	Fit	Resid	Std Resid	
7	32.8	63.6	-30.8	-2.20	R

R Large residual

APPENDIX E: M.A.-TREATED FIBER –DATA AND CENTRAL COMPOSITE DESIGN

StdOrder	RunOrder	PtType	Blocks	Concentra	Time (hrs)	Strength MPa	Modulus MPa	Density g/cm3	Absortion %
1	1	1	1	0	0	183.2071682	1942.880548	1.114382388	92.03747073
2	2	1	1	5	0	109.4728724	821.5020185	1.14954942	58.83575884
3	3	1	1	0	6	116.5127711	941.6927235	1.274434031	69.14285714
4	4	1	1	5	6	108.2634539	1052.684069	1.24729438	63.58381503
5	5	-1	1	-1.03553	3	182.8400287	1039.769577	1.36339986	70.54409006
6	6	-1	1	6.035534	3	159.3071557	1316.551663	1.682456703	70.49808429
7	7	-1	1	2.5	-1.24264	99.72509935	1572.017168	1.848005132	60.7748184
8	8	-1	1	2.5	7.242641	149.9397459	2018.809142	1.344429311	51.02803738
9	9	0	1	2.5	3	159.3018016	2839.928753	1.842462652	56.8627451
10	10	0	1	2.5	3	63.42027819	1565.315202	1.084578456	47.55970924
11	11	0	1	2.5	3	30.07526379	358.7831429	0.4717927	66.2055336
12	12	0	1	2.5	3	35.19507422	578.6765236	0.612994468	55.50510783
13	13	0	1	2.5	3	18.16429587	369.1300743	0.391409888	38.30455259

Central Composite Design

```
Factors:       2     Replicates:     1
Base runs:    13     Total runs:    13
Base blocks:   1     Total blocks:   1

Two-level factorial: Full factorial

Cube points:             4
Center points in cube:   5
Axial points:            4
Center points in axial:  0

α: 1.41421
```

Response Surface Regression: Strength MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	23318.3	4663.7	1.98	0.200
Linear	2	1661.9	831.0	0.35	0.715
Concentration (%)	1	1660.7	1660.7	0.70	0.429
Time (hrs)	1	1.2	1.2	0.00	0.983
Square	2	20584.3	10292.2	4.36	0.059
Concentration (%)*Concentration (%)	1	17582.4	17582.4	7.45	0.029
Time (hrs)*Time (hrs)	1	5129.1	5129.1	2.17	0.184
2-Way Interaction	1	1072.1	1072.1	0.45	0.522
Concentration (%)*Time (hrs)	1	1072.1	1072.1	0.45	0.522
Error	7	16525.8	2360.8		
Lack-of-Fit	3	3399.9	1133.3	0.35	0.796
Pure Error	4	13126.0	3281.5		
Total	12	39844.2			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
48.5884	58.52%	28.90%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		61.2	21.7	2.82	0.026	
Concentration (%)	-28.8	-14.4	17.2	-0.84	0.429	1.00
Time (hrs)	0.8	0.4	17.2	0.02	0.983	1.00
Concentration (%)*Concentration (%)	100.5	50.3	18.4	2.73	0.029	1.02
Time (hrs)*Time (hrs)	54.3	27.2	18.4	1.47	0.184	1.02
Concentration (%)*Time (hrs)	32.7	16.4	24.3	0.67	0.522	1.00

Regression Equation in Uncoded Units

Strength MPa = 169.0 - 52.5 Concentration (%) - 23.4 Time (hrs) + 8.04 Concentration (%) *Concentration (%) + 3.02 Time (hrs)*Time (hrs) + 2.18 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Strength MPa	Fit	Resid	Std Resid	
9	159.3	61.2	98.1	2.26	R

R Large residual

Response Surface Regression: Modulus MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	929007	185801	0.25	0.928
Linear	2	50274	25137	0.03	0.967
Concentration (%)	1	47889	47889	0.06	0.808
Time (hrs)	1	2386	2386	0.00	0.957
Square	2	499049	249524	0.33	0.728
Concentration (%)*Concentration (%)	1	22111	22111	0.03	0.869
Time (hrs)*Time (hrs)	1	442640	442640	0.59	0.468
2-Way Interaction	1	379684	379684	0.51	0.500
Concentration (%)*Time (hrs)	1	379684	379684	0.51	0.500
Error	7	5258070	751153		
Lack-of-Fit	3	667823	222608	0.19	0.895
Pure Error	4	4590247	1147562		
Total	12	6187077			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
866.691	15.02%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		1142	388	2.95	0.021	
Concentration (%)	-155	-77	306	-0.25	0.808	1.00
Time (hrs)	-35	-17	306	-0.06	0.957	1.00
Concentration (%)*Concentration (%)	-113	-56	329	-0.17	0.869	1.02
Time (hrs)*Time (hrs)	504	252	329	0.77	0.468	1.02
Concentration (%)*Time (hrs)	616	308	433	0.71	0.500	1.00

Regression Equation in Uncoded Units

Modulus MPa = 1741 - 109 Concentration (%) - 277 Time (hrs) - 9.0 Concentration (%) *Concentration (%) + 28.0 Time (hrs)*Time (hrs) + 41.1 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Modulus MPa	Fit	Resid	Std Resid	
9	2840	1142	1698	2.19	R

R Large residual

Response Surface Regression: Density g/cm3 versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	0.81960	0.163920	0.62	0.691
Linear	2	0.05217	0.026085	0.10	0.907
Concentration (%)	1	0.02636	0.026363	0.10	0.761

Time (hrs)	1	0.02581	0.025806	0.10	0.764
Square	2	0.76646	0.383229	1.45	0.297
Concentration (%)*Concentration (%)	1	0.36912	0.369124	1.40	0.276
Time (hrs)*Time (hrs)	1	0.49591	0.495906	1.88	0.213
2-Way Interaction	1	0.00097	0.000971	0.00	0.953
Concentration (%)*Time (hrs)	1	0.00097	0.000971	0.00	0.953
Error	7	1.85075	0.264393		
Lack-of-Fit	3	0.40592	0.135307	0.37	0.777
Pure Error	4	1.44483	0.361208		
Total	12	2.67035			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.514192	30.69%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		0.881	0.230	3.83	0.006	
Concentration (%)	0.115	0.057	0.182	0.32	0.761	1.00
Time (hrs)	-0.114	-0.057	0.182	-0.31	0.764	1.00
Concentration (%)*Concentration (%)	0.461	0.230	0.195	1.18	0.276	1.02
Time (hrs)*Time (hrs)	0.534	0.267	0.195	1.37	0.213	1.02
Concentration (%)*Time (hrs)	-0.031	-0.016	0.257	-0.06	0.953	1.00

Regression Equation in Uncoded Units

Density g/cm3 = 1.362 - 0.155 Concentration (%) - 0.192 Time (hrs) + 0.0369 Concentration (%) *Concentration (%) + 0.0297 Time (hrs)*Time (hrs) - 0.0021 Concentration (%) *Time (hrs)

Fits and Diagnostics for Unusual Observations

Obs	Density g/cm3	Fit	Resid	Std Resid	
9	1.842	0.881	0.962	2.09	R

R Large residual

Response Surface Regression: Absortion % versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1338.28	267.66	2.50	0.132
Linear	2	315.88	157.94	1.48	0.292
Concentration (%)	1	188.43	188.43	1.76	0.226
Time (hrs)	1	127.45	127.45	1.19	0.311
Square	2	831.38	415.69	3.89	0.073
Concentration (%)*Concentration (%)	1	802.26	802.26	7.50	0.029
Time (hrs)*Time (hrs)	1	81.80	81.80	0.76	0.411
2-Way Interaction	1	191.03	191.03	1.79	0.223
Concentration (%)*Time (hrs)	1	191.03	191.03	1.79	0.223
Error	7	748.85	106.98		
Lack-of-Fit	3	307.78	102.59	0.93	0.504
Pure Error	4	441.07	110.27		
Total	12	2087.14			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
10.3431	64.12%	38.49%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		52.89	4.63	11.43	0.000	

Concentration (%)	-9.71	-4.85	3.66	-1.33	0.226	1.00
Time (hrs)	-7.98	-3.99	3.66	-1.09	0.311	1.00
Concentration (%)*Concentration (%)	21.48	10.74	3.92	2.74	0.029	1.02
Time (hrs)*Time (hrs)	6.86	3.43	3.92	0.87	0.411	1.02
Concentration (%)*Time (hrs)	13.82	6.91	5.17	1.34	0.223	1.00

Regression Equation in Uncoded Units

Absortion % = 82.81 - 13.30 Concentration (%) - 5.92 Time (hrs) + 1.718 Concentration (%) *Concentration (%) + 0.381 Time (hrs)*Time (hrs) + 0.921 Concentration (%) *Time (hrs)

APPENDIX F: NAOCL-TREATED FIBER –DATA AND CENTRAL COMPOSITE DESIGN

StdOrder	RunOrder	PtType	Blocks	Concentra	Time (hrs)	Strength MPa	Modulus MPa	Density g/cm3	Absortion %
1	1	1	1	0	0	183.2071682	1942.880548	1.114382388	92.03747073
2	2	1	1	10	0	133.8498601	940.0031651	1.104432304	55.64202335
3	3	1	1	0	6	83.16182463	444.1959873	0.965526129	81.74962293
4	4	1	1	10	6	32.59670435	186.6197241	0.416706951	85.80542265
5	5	-1	1	-2.07107	3	182.2051834	981.8534915	1.323432184	79.15690867
6	6	-1	1	12.07107	3	81.92869505	303.5157522	0.531998596	90.48543689
7	7	-1	1	5	-1.24264	210.5475831	828.1099371	1.770058643	91.05613701
8	8	-1	1	5	7.242641	62.40146626	217.6441086	0.56619504	72.11428571
9	9	0	1	5	3	255.1129566	1084.588024	2.366629093	66.37931034
10	10	0	1	5	3	127.8088867	599.8719784	1.104484198	61.0989011
11	11	0	1	5	3	135.7413931	527.315178	1.019522005	49.49402024
12	12	0	1	5	3	159.7301309	901.6762946	1.365946932	65.91928251
13	13	0	1	5	3	52.45989122	320.9665179	0.565350584	55.30612245

Central Composite Design

```
Factors:       2     Replicates:     1
Base runs:    13     Total runs:    13
Base blocks:   1     Total blocks:   1

Two-level factorial: Full factorial

Cube points:             4
Center points in cube:   5
Axial points:            4
Center points in axial:  0

α: 1.41421
```

Response Surface Regression: Strength MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	13399.9	2679.98	0.52	0.757
Linear	2	174.8	87.39	0.02	0.983
Concentration (%)	1	134.0	133.97	0.03	0.877
Time (hrs)	1	40.8	40.81	0.01	0.932
Square	2	6471.5	3235.77	0.63	0.562
Concentration (%)*Concentration (%)	1	675.6	675.61	0.13	0.728
Time (hrs)*Time (hrs)	1	5197.0	5197.01	1.00	0.350
2-Way Interaction	1	6753.6	6753.58	1.31	0.291
Concentration (%)*Time (hrs)	1	6753.6	6753.58	1.31	0.291
Error	7	36203.9	5171.99		
Lack-of-Fit	3	8891.2	2963.75	0.43	0.741
Pure Error	4	27312.7	6828.17		
Total	12	49603.8			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
71.9165	27.01%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		145.9	32.2	4.54	0.003	
Concentration (%)	-8.2	-4.1	25.4	-0.16	0.877	1.00
Time (hrs)	4.5	2.3	25.4	0.09	0.932	1.00
Concentration (%)*Concentration (%)	19.7	9.9	27.3	0.36	0.728	1.02

Time (hrs)*Time (hrs)	-54.7	-27.3	27.3	-1.00	0.350	1.02
Concentration (%)*Time (hrs)	82.2	41.1	36.0	1.14	0.291	1.00

Regression Equation in Uncoded Units

Strength MPa = 171.4 - 13.0 Concentration (%) + 10.6 Time (hrs) + 0.39 Concentration (%) *Concentration (%) - 12.1 Time (hrs)*Time (hrs) + 5.48 Concentration (%) *Time (hrs)

Response Surface Regression: Modulus MPa versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1220278	244056	0.91	0.525
Linear	2	406063	203031	0.76	0.504
Concentration (%)	1	181662	181662	0.68	0.438
Time (hrs)	1	224401	224401	0.84	0.391
Square	2	123914	61957	0.23	0.800
Concentration (%)*Concentration (%)	1	17	17	0.00	0.994
Time (hrs)*Time (hrs)	1	121411	121411	0.45	0.523
2-Way Interaction	1	690301	690301	2.57	0.153
Concentration (%)*Time (hrs)	1	690301	690301	2.57	0.153
Error	7	1877782	268255		
Lack-of-Fit	3	785268	261756	0.96	0.494
Pure Error	4	1092513	273128		
Total	12	3098060			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
517.933	39.39%	0.00%	0.00%

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		855	232	3.69	0.008	
Concentration (%)	-301	-151	183	-0.82	0.438	1.00
Time (hrs)	-335	-167	183	-0.91	0.391	1.00
Concentration (%)*Concentration (%)	3	2	196	0.01	0.994	1.02
Time (hrs)*Time (hrs)	-264	-132	196	-0.67	0.523	1.02
Concentration (%)*Time (hrs)	831	415	259	1.60	0.153	1.00

Regression Equation in Uncoded Units

Modulus MPa = 1458 - 114 Concentration (%) - 212 Time (hrs) + 0.06 Concentration (%) *Concentration (%) - 58.7 Time (hrs)*Time (hrs) + 55.4 Concentration (%) *Time (hrs)

Response Surface Regression: Density g/cm3 versus Concentration (%), Time (hrs)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	0.43996	0.087992	0.41	0.827
Linear	2	0.04220	0.021099	0.10	0.907
Concentration (%)	1	0.03608	0.036084	0.17	0.694
Time (hrs)	1	0.00611	0.006114	0.03	0.871
Square	2	0.30905	0.154524	0.72	0.518
Concentration (%)*Concentration (%)	1	0.17874	0.178737	0.84	0.391
Time (hrs)*Time (hrs)	1	0.09166	0.091663	0.43	0.534
2-Way Interaction	1	0.08871	0.088714	0.41	0.540
Concentration (%)*Time (hrs)	1	0.08871	0.088714	0.41	0.540
Error	7	1.49691	0.213845		
Lack-of-Fit	3	0.41675	0.138917	0.51	0.694
Pure Error	4	1.08016	0.270040		
Total	12	1.93687			

Model Summary

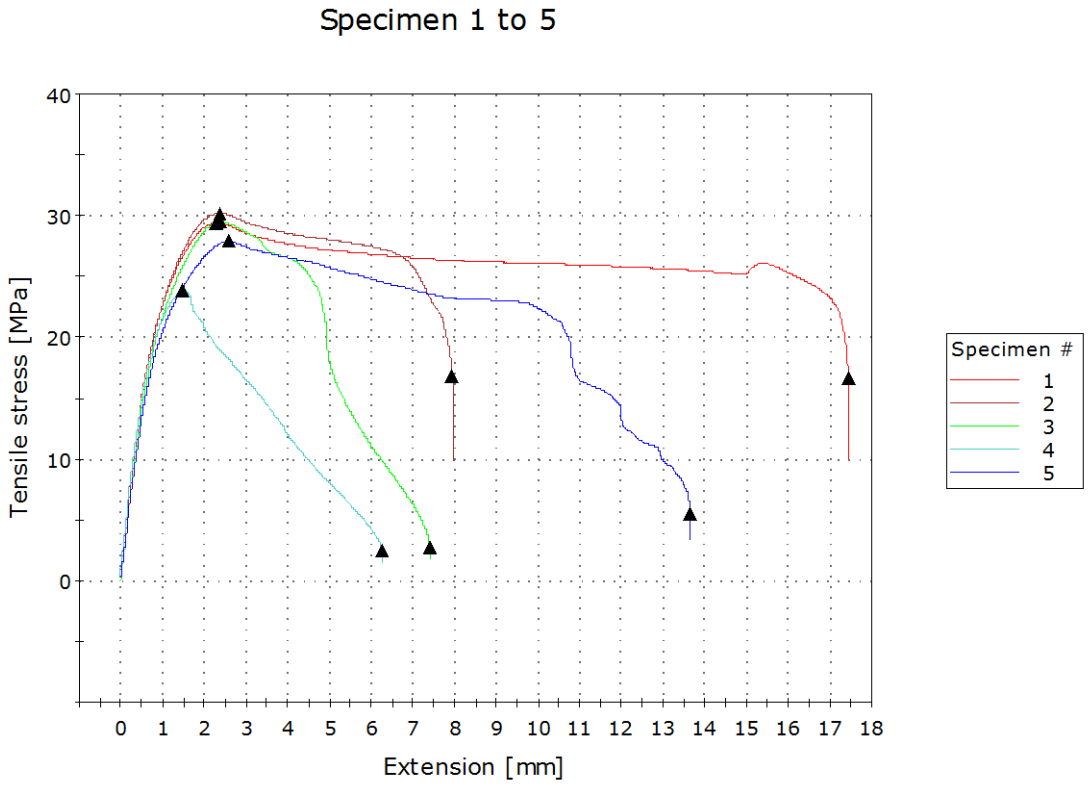
```
       S    R-sq  R-sq(adj)  R-sq(pred)
0.462433  22.71%      0.00%       0.00%


Coded Coefficients

Term                                  Effect    Coef  SE Coef  T-Value  P-Value   VIF
Constant                                         1.004    0.207     4.86    0.002
Concentration (%)                     -0.134  -0.067    0.163    -0.41    0.694  1.00
Time (hrs)                            -0.055  -0.028    0.163    -0.17    0.871  1.00
Concentration (%)*Concentration (%)    0.321   0.160    0.175     0.91    0.391  1.02
Time (hrs)*Time (hrs)                 -0.230  -0.115    0.175    -0.65    0.534  1.02
Concentration (%)*Time (hrs)           0.298   0.149    0.231     0.64    0.540  1.00


Regression Equation in Uncoded Units

Density g/cm3 = 1.293 - 0.1073 Concentration (%) + 0.035 Time (hrs)
                + 0.00641 Concentration (%)*Concentration (%) -
 0.0510 Time (hrs)*Time (hrs)
                + 0.0199 Concentration (%)*Time (hrs)
```

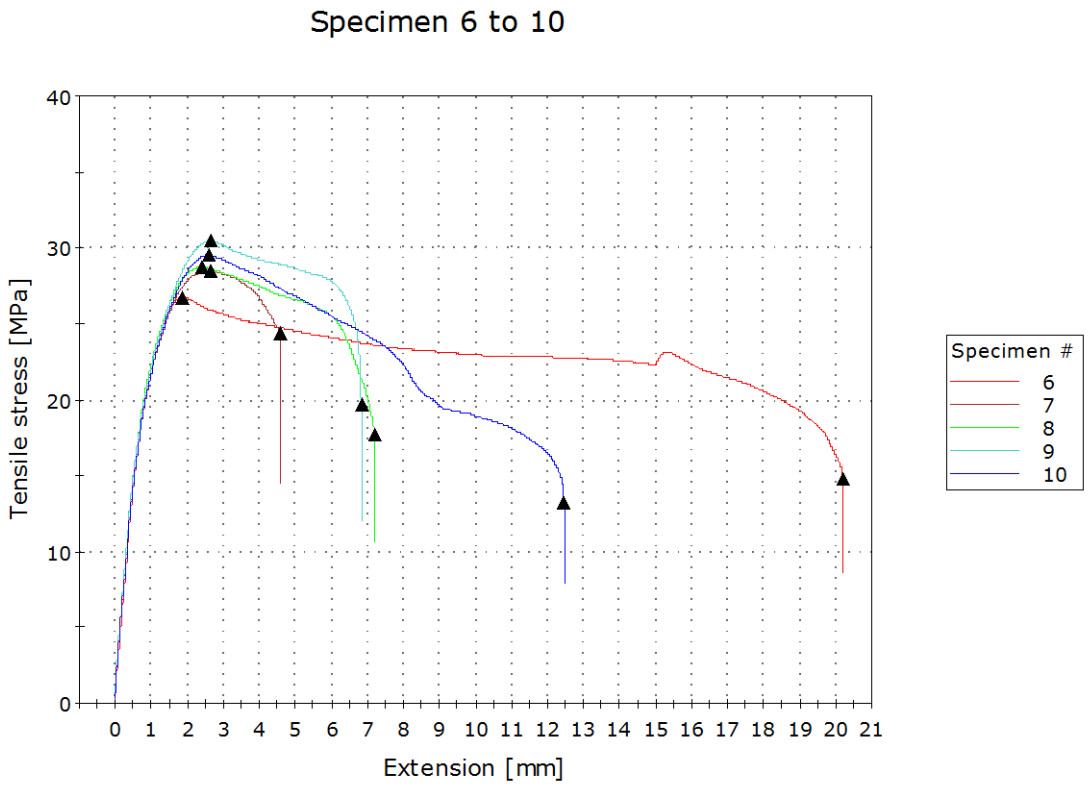
Response Surface Regression: Absortion % versus Concentration (%), Time (hrs)

```
Analysis of Variance

Source                                  DF   Adj SS   Adj MS  F-Value  P-Value
Model                                    5   529.63  105.926     1.02    0.471
  Linear                                 2   223.61  111.805     1.08    0.391
    Concentration (%)                    1   223.52  223.518     2.16    0.185
    Time (hrs)                           1     0.09    0.093     0.00    0.977
  Square                                 2   304.01  152.004     1.47    0.294
    Concentration (%)*Concentration (%)  1   285.66  285.662     2.76    0.141
    Time (hrs)*Time (hrs)                1     4.17    4.170     0.04    0.847
  2-Way Interaction                      1     2.01    2.010     0.02    0.893
    Concentration (%)*Time (hrs)         1     2.01    2.010     0.02    0.893
Error                                    7   725.68  103.668
  Lack-of-Fit                            3   155.14   51.712     0.36    0.785
  Pure Error                             4   570.54  142.635
Total                                   12  1255.31


Model Summary

      S    R-sq  R-sq(adj)  R-sq(pred)
10.1818  42.19%      0.90%       0.00%


Coded Coefficients

Term                                  Effect   Coef  SE Coef  T-Value  P-Value   VIF
Constant                                      76.62     4.55    16.83    0.000
Concentration (%)                    -10.57  -5.29     3.60    -1.47    0.185  1.00
Time (hrs)                             0.22   0.11     3.60     0.03    0.977  1.00
Concentration (%)*Concentration (%)   12.82   6.41     3.86     1.66    0.141  1.02
Time (hrs)*Time (hrs)                 -1.55  -0.77     3.86    -0.20    0.847  1.02
Concentration (%)*Time (hrs)          -1.42  -0.71     5.09    -0.14    0.893  1.00


Regression Equation in Uncoded Units

Absortion % = 86.73 - 3.48 Concentration (%) + 1.58 Time (hrs) + 0.256 Concentration (%)
              *Concentration (%) - 0.34 Time (hrs)*Time (hrs) - 0.095 Concentration (%)
              *Time (hrs)


Fits and Diagnostics for Unusual Observations

                                  Std
Obs  Absortion %    Fit  Resid  Resid
 10        97.44  76.62  20.82   2.29  R

R  Large residual
```

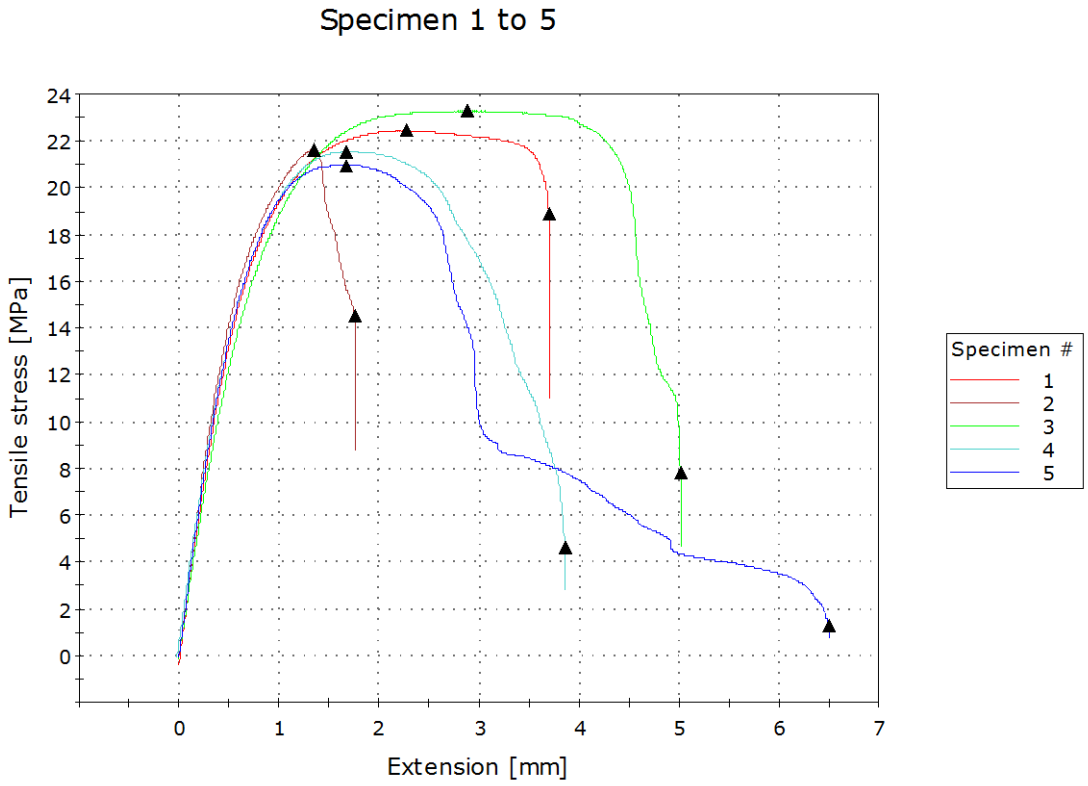
APPENDIX G: TENSILE PLOTS OF POLYPROPYLENE AND COMPOSITE SAMPLES

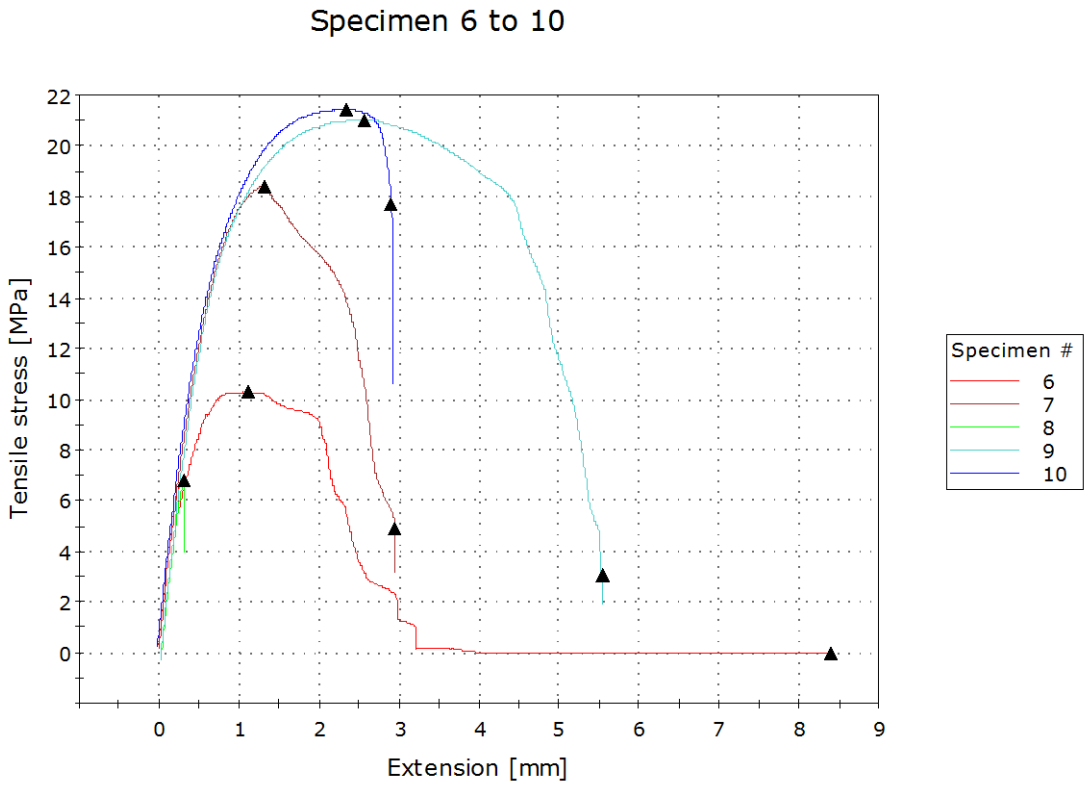
POLYPROPYLENE >>



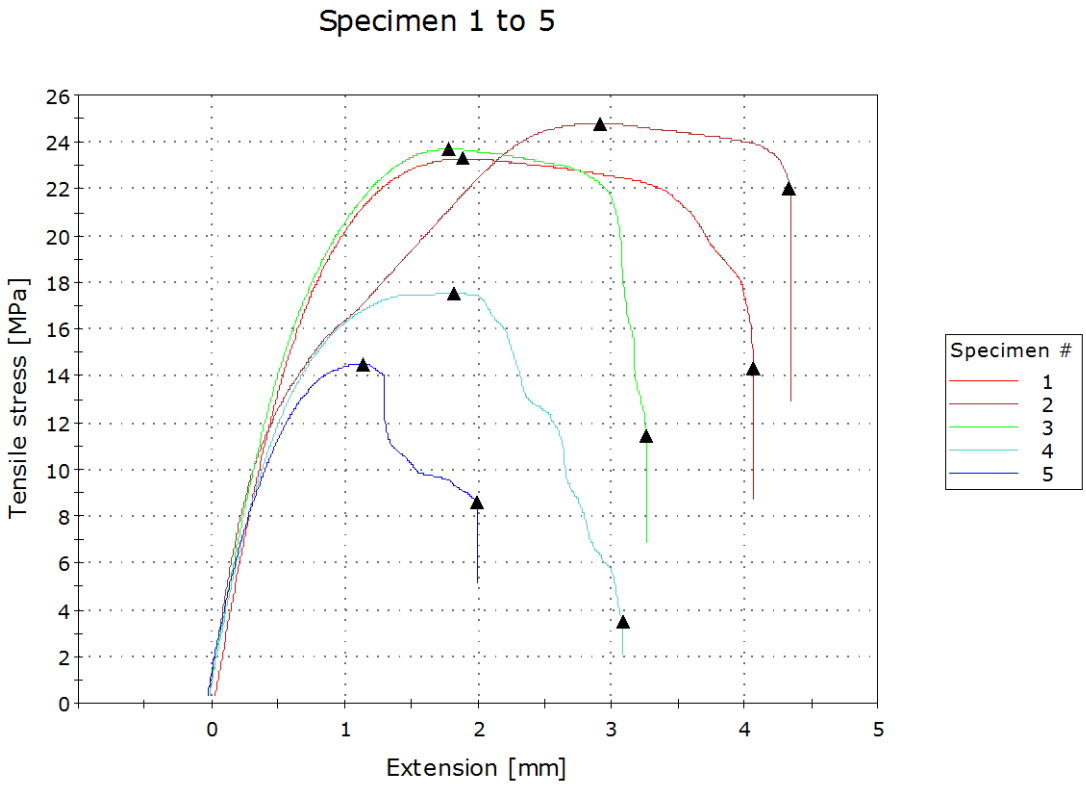


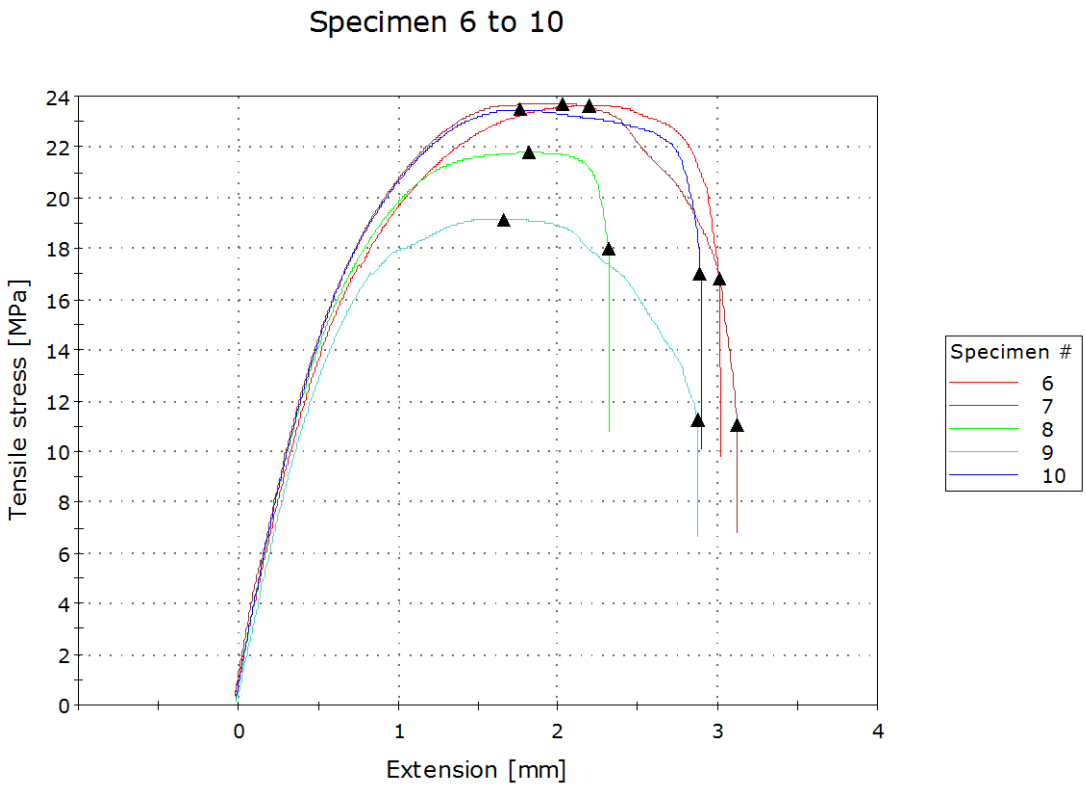
RAW-FIBER COMPOSITES>>





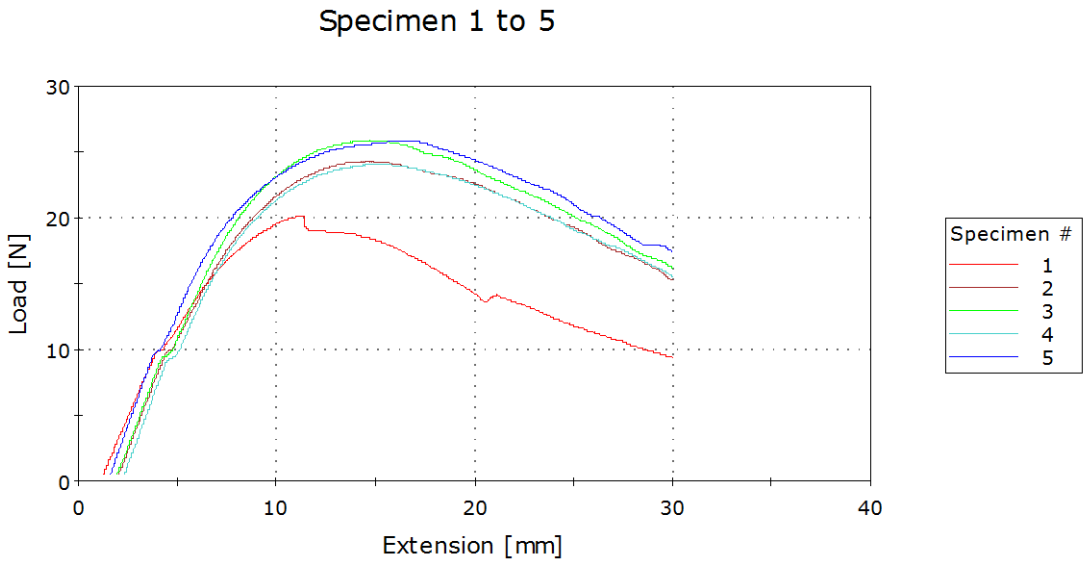
TREATED-FIBER COMPOSITES>>

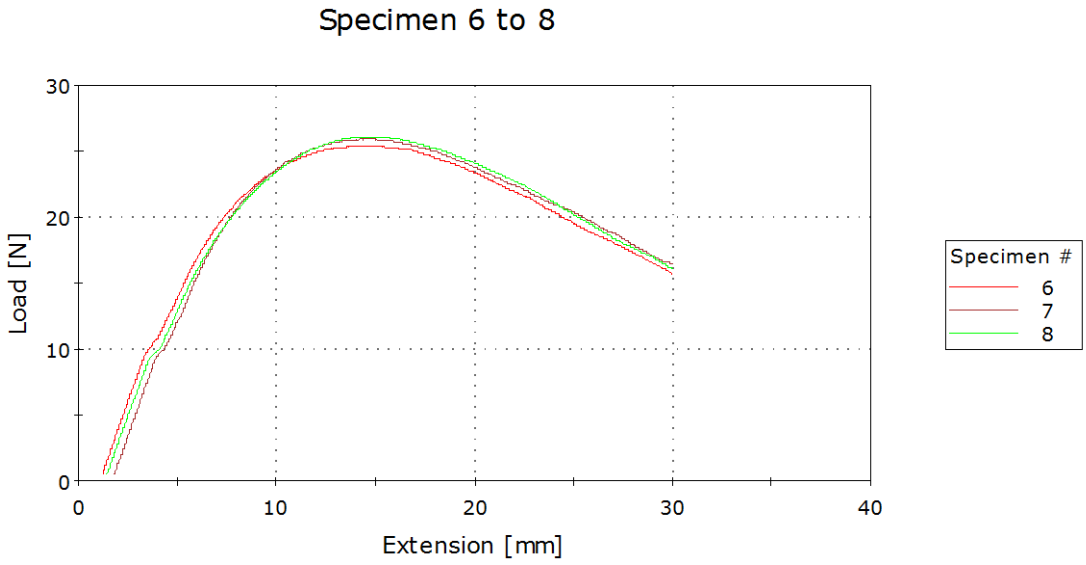




APPENDIX H: BENDING PLOTS OF POLYPROPYLENE AND COMPOSITE SAMPLES

POLYPROPYLENE >>





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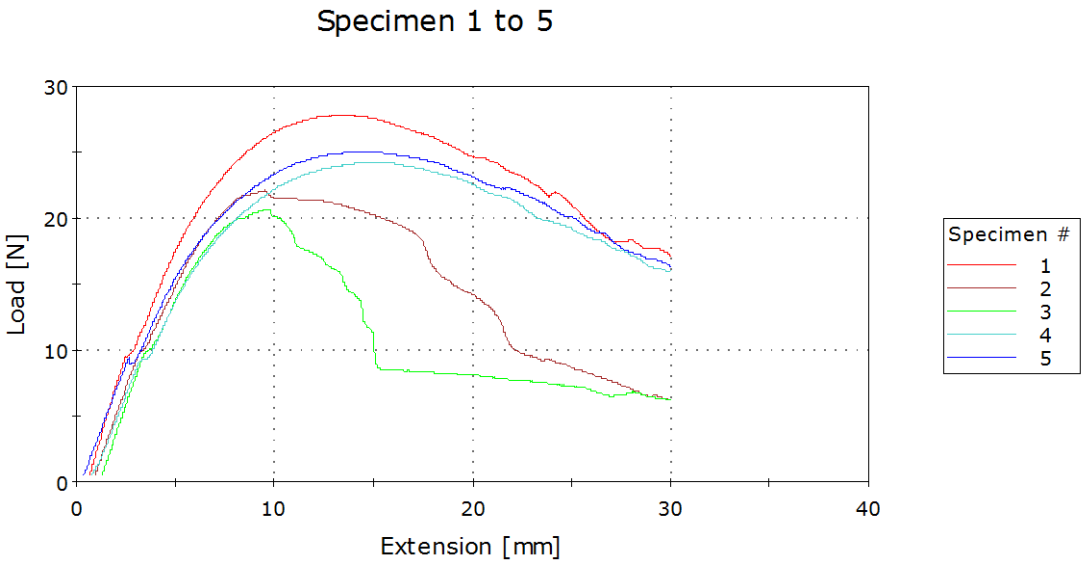

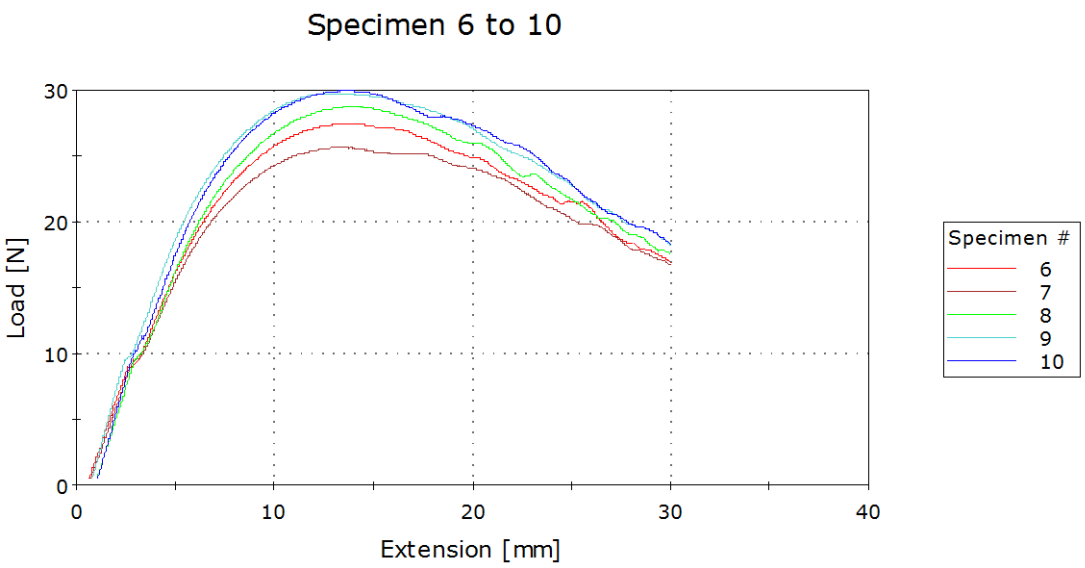

TREATED-FIBER COMPOSITES>>